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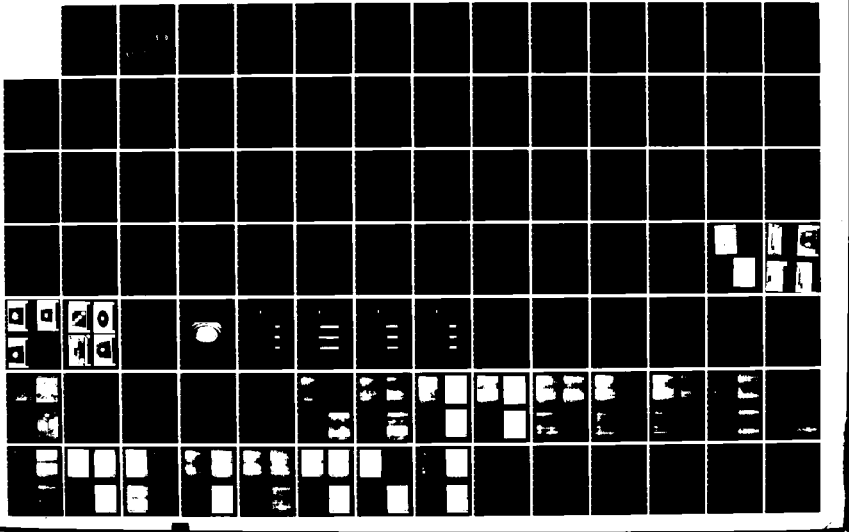

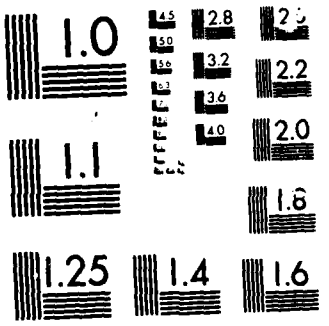

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PRODUCTION PROVE-OUT OF A PROCESS FOR REMELTING DEPLETED URANIUM MACHINING CHIPS BY VACUUM INDUCTION

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Depleted Uranium
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20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

An MM&T program to pursue a production prove-out of recycling depleted uranium machining chips in an existing vacuum induction remelting system was conducted. A viable method of processing DU chips and generating briquettes for remelting was developed. A pilot lot of 80 finished, machined M833 penetrators, fabricated under the guidelines of this program, is available for ballistic testing. Included in this report is a general analysis of facility requirements to implement chip recycling into the Aerojet production cycle.

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INTRODUCTION

The objective of this program was to determine the feasibility of introducing DU-3/4 wt.% Ti machining chips in the form of compacted briquettes into the vacuum remelting process stream for large caliber core penetrators.

Briquetting technology was pre-selected as the recycling method as it was the least costly process technology which could be used in conjunction with currently available production process equipment at Aerojet Ordnance Co.

The possible significant reduction in waste handling and disposal, both in physical as well as monetary terms, makes chip recycling an attractive alternative. However, the effects of chip briquettes recycled into the process stream must be assessed in terms of the final quality and performance of the end product. In addition, the effects of various chip recycling process parameters must be evaluated. The cost of any chip recycling process must be ultimately weighed against those associated with the disposal of the chips.

SCOPE

This investigation was divided into three technical tasks. Task A, *Chip/Briquette Processing*, was a study of the parameters associated with the processing of chips into briquettes themselves. This work included an evaluation of chip cleaning and crushing, briquette compaction, and studies of chip and briquette in-process storage. An important common thread throughout all Task A work was the effect of chip particle size distribution. From this work, an optimum chip particle size distribution for briquetting was determined for use in Task B and Task C work.

Task B, Remelt Briquette-Charge Effects, involved the analysis of the effects of introducing different quantities of briquettes on billet chemistry and quality. The work involved extensive chemical and metallographic analysis on the four trial melt-heats which were produced.

The fabrication of large caliber core material utilizing chip recycling technology was undertaken as Task C. A pilot lot of M833 penetrators was fabricated from a melt-heat of material which had included chip briquettes in the remelt charge. The briquettes used for this remelt charge were made using the optimum particle size distribution determined as part of Task A. The quantity of briquettes in the charge was the optimum determined as part of Task B. The subsequent processing of the melt-heat into M833 cores was performed using normal production procedures for all operations. The melt-heat was evaluated for mechanical properties and all blanks were inspected ultrasonically and for surface hardness again in accordance with normal procedures.

TASK A - CHIP/BRIQUETTES PROCESSING

Task A's objective was the determination of the significant parameters for processing DU-3/4 wt.%Ti machining chips into briquettes suitable for remelting in the VIR process. Five different combinations of coarse OD turnings and fine finish turnings were examined for the following factors:

1. Determination of typical foreign material with chips
2. Nitric acid chip surface cleaning
3. Processing yields up to briquetting
4. Effect of processing on chip chemistry
5. Chip storage life

In addition, factors pertinent to briquetting and their handling and storage characteristics were determined.

Procedures

All chips studied in Task A were processed in a common manner. Chips were collected from the two basic machining operations characteristic of large caliber core production, coarse OD turning and fine finish machining. Chips were gathered from the collection barrels on each lathe. The collection barrels were fed by conveyers transporting chips and coolant from the lathe bed. All foreign material dropped into the lathe will find its way into the collection system's chips.

The collected chips must be inspected for foreign material and this material removed. Chips were inspected while the coarse and fine fractions were weighed and blended. Chips were also scrutinized while they were loaded in the ring crusher.

Figure 1 shows the basic chip processing procedure. Samples of as-collected chips were submitted for chemical analysis. Five 150 pound batches of chips were weighed up in the portions shown in Table 1. Chips were blended by stirring with a rake in the barrel. The long, tangled chip strands were then crushed in the ring crusher (see Figures 2 and 3) and collected in a coolant filled barrel below the crusher. Samples of crushed chips were submitted for chemical analysis.

Crushed chips were removed from the coolant and sieved over a 12 mesh screen to remove fine chips and thus reduce the fire hazard. After sieving, all five chip batches were weighed to determine losses to crushing and screening. The remaining chips (larger than 12 mesh) were loaded into stainless steel mesh baskets and washed in an ionic detergent solution for three minutes. The detergent removed the lathe machining lubricant. The basket of chips was then transferred to a water rinse tank for three more minutes. After rinsing, the basket of chips was air dried in a centrifugal dryer for 10 minutes.

The cleaned chips were then pickled in a nitric acid bath to remove surface oxides for both cleanliness and enhanced compactability to the briquette form. After pickling in the nitric acid solution, the chips

were rinsed in a water tank to remove nitric acid from the chips' surface. Finally, the wet chips were air dried for 10 minutes in the centrifugal dryer again.

Each batch of chips was then weighed for post-pickling yield and then split into halves. One half was stored in a closed-air environment, while the other half was stored in a closed, cold CO_2 environment to inhibit oxidation.

Samples were taken periodically from each storage system for two weeks to determine qualitatively the oxidation rate. These samples were photographed to show the degree of oxidation.

The bulk density of loose crushed and sized machining chips is too low to permit efficient remelt furnace loading. Accordingly, the chips were briquetted using a 4140 flame hardened chrome plated steel punch and a D-2 tool steel die on a 300 T Dake press. Chips were pressed in six pound batches, resulting in 3-7/8" diameter by 1-1/2" tall briquettes. The chips were loaded in the 6" tall steel die resting on the 300 ton Dake press. Approximately four pounds of chips were loaded in the die for a 100 ton pre-press. This allowed the remaining two pounds to be put in the die for the final 300 ton (51,000 psi) pressing. Six briquettes were pressed from each of the five batches.

Each compacted briquette was weighed to determine the percent theoretical density, the ratio of measured briquette density to DU-3/4 wt.%Ti's theoretical density. Briquettes were also evaluated for surface texture and handlability.

Briquettes from each batch were stored in air and cold CO_2 for up to two months. Periodic inspections were made to note surface oxidation and handlability.

Results and Discussion

Machining chips were collected and blended in five groups as shown in Table 1. Inspection showed the presence of a number of contaminants that are listed below:

1. Tungsten carbide tool inserts
2. High speed steel center drills
3. Tool insert cardboard and plastic packaging
4. Steel allen wrenches
5. Steel screws - 1/4" diameter
6. Paper towels
7. Rubber gloves
8. Solid chunks of DU from the sampling lathe
9. Other miscellaneous items

A number of these contaminants could have caused damage to processing equipment, particularly the ring crusher. This damage could have come in two forms. First, the tool inserts, center drills, allen

wrenches, and solid chunks could damage the ring crusher and cause accelerated wear. The second and more serious danger was the potential of fire. Any of the above contaminants would be prone to cause sparks which could easily ignite the DU chips which are crushed dry.

Besides equipment and safety considerations, remelt heat chemical analysis could be threatened by the contaminants. The center drills, allen wrenches, steel screws, and analog stainless steel turnings could significantly raise the iron concentration in the remelt heat chemical analysis. Analog materials such as stainless steel and aluminum are used to fabricate "clean" penetrators. Clean penetrators may be used to check handling equipment at the vendor's plant or for demonstration purposes. Other contaminants such as the rubber gloves and the paper towels could signficantly raise carbon levels of a remelt heat.

While a manual inspection procedure was used in this project, contaminants could be removed from a production process by a combination of visual and magnetic separation. Visual inspection would result in removal of items like paper towels, rubber gloves, tool insert packaging, and other miscellaneous material.

Magnetic separation would remove the magnetic materials such as allen wrenches, screws, and other tools or scrap. Tungsten carbide tool inserts also might be removed by magnetic separation techniques since the cobalt binder is paramagnetic.

The chip cleaning in nitric acid was straightforward. Initial pickling trials were run for several chip mixes and pickling conditions as shown in Table 2. Nitric acid pickling bath temperature, the normality of the nitric acid pickling solution and the overall chip surface area determined how quickly and how well uranium oxide was removed from the DU 3/4 wt/o Ti chips. Pickling bath temperature was the most significant factor affecting the reaction kinetics of uranium oxide removal from chips. The preliminary results shown in Table 2 were confirmed on large size batches. At low temperatures, between 60^{o}F and 85^{o}F, pickling 150 pound batches of chips required 40 minutes. Higher temperatures, between 90^{o}F and 110^{o}F, required only 20 minutes to remove the oxide from the chips. Temperatures above 110^{o}F cleaned the chip surface in less than 10 minutes.

However, increasing oxide removal rates associated with higher temperatures led to increased nitrous oxide emissions. Temperatures above 90^{o}F generated rapid evolution of nitrous oxide which taxed the pickle tank's scrubber capacity. When bath temperatures reached 110^{o}F, the nitrous oxide evolution was so rapid that the fumes exceeded the ventilation system's capacity. Pickling temperatures were carefully regulated to optimize oxide removal, while minimizing the rapid release of toxic nitrous oxide.

Chip oxide removal rate was also dependent on the nitric acid solution normality. Higher normality acid solutions yielded more rapid and more complete oxide removal. More concentrated nitric acid solutions also produced a more rapid generation of nitrous oxide fumes.

The loss of chips through the grinding and cleaning process is an important process parameter because it is an important cost consideration. Table 3 shows the yields of the various chip size distribution batches through the chip processing steps. Batch 1 was made up entirely of coarse OD turnings and even after crushing showed larger chips than the other batches. Larger chip sizes resulted in a much lower fallout of fines during sieving. Batch 1 showed a 4% weight loss to crushing and sieving while the next lowest weight loss was 19% associated with virtually all the remaining chip test groups. Batch 1 also had the lowest weight loss due to pickling. This was attributed to the relatively low surface area to volume ratio associated with these chips. The net chip processing yield for Batch 1 was 86%. Addition of fine machining chips appears to increase both the screen and pickling losses. It would appear that a mix of chips representing time of production could be expected to lose about 30-35% of the initial chip weight to screening and pickling.

The chip processing did not affect chip chemistry. Table 4 shows the results of chemical analysis of both coarse and fine chips both before and after crushing and pickling. Overall, there was very little variation in chemistries between the processing steps. Virtually all values lay within the normal analytical scatter for each element. No element showed an increase of more than 5 ppm between the unprocessed analysis and the post pickling analysis, making it impossible to differentiate between increases and scatter. Lead was picked up by the chips and remained at constant concentrations throughout the processing. Lead levels were higher in the OD chips. OD chips are the first rough turnings after blanks are aged in the lead pot. Accordingly, one might expect OD chips to exhibit more residual lead than the finishing chips. Even these low lead concentrations (under 11 ppm) posed no problem in remelt.

Table 4 also shows a minor variation in chemistries with regard to hydrogen concentrations. Coarse chips of Batch 1 showed a 0.8 ppm hydrogen concentration, while the fine chips of Batch 5 showed a 2.1 ppm. The higher reported hydrogen for the fine chips was attributed to their much greater surface area. Significantly, there was no evidence of iron pick-up in these experiments.

Storage of processed chips may be required in some production situations. Accordingly, chips were stored in both plant air and cold CO_2 within closed containers. Figures 4 through 10 show the chips stored in open air and under dry ice. The cleaned DU-3/4 wt.%Ti chips oxidized from a medium gold color to a dark gold or medium brown color within two days. Oxidation after this proceeded at a very slow rate due to the relatively protective oxide layer on the surface of the chips. Dry ice did not appear to retard or accelerate the oxidation process. Two week old chips could be pressed into briquettes without any hindrance from the oxide layer.

It should be noted that crushed, sized and cleaned chips did not spontaneously ignite at any time, even during relatively long storage in closed containers containing plant air.

Briquettes were readily fabricated from the cleaned DU-3/4 wt.%Ti chips. Table 5 shows how the briquettes from each batch rated in terms of quality. All five batch types, ranging from coarse to fine chips, made good quality briquettes which could be readily handled without fear of breaking up.

There was some variance in the bulk density (% theoretical density) of the six briquettes pressed in each batch. The actual briquette volume was estimated since the briquette's raised beveled upper rim was not that of a true cylinder. Also, small height variations (under 0.1 inches) existed between briquettes; internal chip batch variations were minimized by averaging the densities of six briquettes from each batch type.

Batch 1's 100% coarse OD chips gave the lowest percent theoretical density, 40.9%. The remaining batches of briquettes had percent theoretical densities between 43 and 45%.

Batch 1's 100% coarse chips gave a coarse briquette surface texture. Each individual chip was readily discernable in the briquette surface and small voids could be detected between some of the surface chips. The voids did not noticeably effect the overall briquette's integrity, but did allow some spalling of chips from the top and bottom briquette surfaces. The remaining four chip batches contained fine chips which allowed better consolidation and a corresponding smoother surface texture. Voids between chips on the briquette surfaces were much smaller. Smoother surface texture eliminated spalling from the top and bottom briquette surfaces on the remaining four batches.

DU 3/4 wt/o Ti briquettes stored very well in both air and in a dry ice environment. Briquettes stored for over two months still had the dark gold to light brown color associated with good briquettes. Figures 11-14 show briquettes immediately after pressing, after 5 days, after 7 days, and after 14 days. Briquettes oxidized most rapidly the first two days in both air and dry ice environments. After two days, the briquettes turned a dark gold as compared to the medium gold freshly pressed briquettes. Figures 11, 12, 13, and 14 illustrated the rather stable surface oxide color over two weeks of observation. The briquettes shown in Figures 12-14 were stored in dry ice. Briquettes stored in open air showed the same qualitative color change.

DU 3/4 wt/o Ti chip briquettes stored readily in open air a period of several months. Further study would be needed to determine the maximum DU 3/4 wt/o Ti briquette life. While the briquettes looked good after two months, they were not used in a remelt heat.

The results of detailed chemical analyses of thirty-six locations on one ingot of each heat are given in Tables 9, 10, 11, and 12. Due to the large amount of analysis data, the radial values were averaged for each of the twelve slices. Similarly, the longitudinal values were averaged for each ingot. Tables 13, 14, 15, and 16 show average chemistry variations radially, while Tables 17, 18, 19, and 20 show average chemistry variations longitudinally.

Iron, silicon, carbon, and lead were emphasized in this study due to their known or suspected deleterious effect on properties. The data for these elements from the tables is also presented graphically in Figures 17-24.

There were no patterns of radial segregation in the billets of each heat as shown in Figures 17-20. OD, mid-radius and centerline values were consistent when one considered the standard deviation associated with each group.

Also, the longitudinal chemical analysis data showed no segregation patterns for iron, silicon and carbon in any of the four heats, Figures 21-24. Some unexplained longitudinal lead segregation was noticed in heat 5056, the control heat, and heat 5130, the 15% chip briquette heat, Figure 23.

There is no specified limit for lead in the M833 chemistry specification. Lead concentrations of 20 ppm are low and have no detrimental effect on the mechanical properties or processing of M833 cores.

Figures 17 and 21, radial and longitudinal Fe chemistry, show the high iron content mentioned above for heat 5245 (20% chips). Both the detailed billet study and the production analysis were done on the same billet. The difference in values, 92 ppm for the detailed analysis and 56 ppm for the production analysis, can be explained by an analysis of the relative sensitivity of the spectrographic calibration curve for iron. The calibration curve for iron is given in Figure 25. The threshold limit for iron is around 19-20 ppm; between 19 ppm and 50 ppm iron (the maximum allowed in M833) there is high analysis sensitivity as the figure shows. However, above 50 ppm the sensitivity decreases rapidly and small changes in signal intensities will be read as large changes in iron concentration. Accordingly, high iron values (above about 50-60 ppm) are difficult to accurately determine. The high standard deviation values for heat 5245 from Table 16 and Table 20 reflect this spectrographic insensitivity to high iron concentration.

Task B heats were analyzed for microcleanliness, the presence of inclusions and second phases in the microstructures. Micrographs taken at 100X showed the production and chip heats to be comparable in microcleanliness. The micrographs are shown in Appendix A. A point intercept analysis of each sample showed quantitatively that chip remelt

Summary

The chip and briquette processing procedures recommended in the original contract proposal worked quite well. This involved sorting and blending the OD and finish lathe turnings to the desired proportions. Chips were then crushed dry in the ring crusher. Crushed chips were screened on a 12 mesh sieve. Fine chips fell through the screen and the plus 12 mesh chip fraction was washed in an ionic detergent solution and rinsed in water. The rinsed chips were air dried for 10 minutes in the centrifugal dryer. The dried chips were pickled in a nitric acid solution for a period of time dependent on the pickle bath temperature and rinsed in water. Wet pickled chips were dried for ten minutes in centrifical dryer. The dried, cleaned chips were stored either in air or under dry ice. Chips were pressed on the Dake press using the full 300 ton pressure. Briquettes were stored either in air or under dry ice until they were weighed as a remelt furnace charge.

TASK B - REMELT BRIQUETTE - CHARGE EFFECTS

Task B had three major objectives. The first was to produce heats with varying quantities of chips to determine the casting feasibility. Characteristics such as melt pool behavior, residual within the crucible, billet surface quality and routine ingot chemistry were examined. The second was to determine the effect, if any, of remelt chips on the ingot macrosegregation. Finally, the third was to compare the ingot microcleanliness of chip remelt heats to production heats. Figure 15 shows the overall experimental structure of Task B.

Procedure

Four remelt heats were cast for Task B. These included a control heat, a 5% chip heat, a 15% chip heat and a 20% chip heat. The chips were collected and processed into briquettes containing 33% coarse and 67% fine chips. Figure 16 shows how a 15% briquette charge filled the crucible. Briquettes were loaded on top of the total charge for all chip recycle heats. Standard remelt procedures were used and all heats were produced in the same furnace to preclude furnace variables affecting the results. The melt down of the charge was carefully observed in order to determine the effect of chips on the VIR process. The melted charge was bottom poured into standard production ingot molds with no special treatment. After the cast ingots were broken out of the molds, they were studied for surface quality. Table 6 gives the remelt charge for each of these heats. As can be seen, all four heats utilized process scrap and two grade A derbies.

Standard M833 specification chemical analysis was performed on the A ingot of each heat. Samples were taken from the top and bottom of each ingot of the heat for additional titanium and carbon values.

Following the normal production chemical analysis, the A ingot of each heat was cut transversly into 12 equal sections. Each section was analyzed at the OD, mid-radius and centerline location to determine potential chemistry variations within the billet caused by the use of recycle chips.

Each of these heats was examined for microcleanliness. Top and bottom transverse ingot slices were further sectioned at the OD, mid-radius and centerline locations. Each sample was carefully mounted in bakelite to retain orientation. Mounted samples were metallographically polished for examination in the JEOL 733 scanning electron microscope at 100X. The scanning electron microscope (SEM) was chosen because it provides superior contrast between the DU matrix and second phases.

The second phases present were identified using the EDS and WDS chemical analysis which determined elements present by studying x-rays emitted from the material in the SEM. EDS, energy dispersive system of the SEM, utilizes the relative energy intensity of x-rays emitted from

the sample to identify the element focused on. EDS can identify elements of the matrix and second phase to atomic weights as low as Berylium. Elements with lower atomic weights can be verified with the WDS, wavelength dispersive system, on the SEM. WDS utilizes the traditional x-ray diffraction technique where a known wavelength of emitted x-rays is diffracted with a crystal to identify lighter elements such as carbon.

A point intercept technique was used to quantitatively determine the microcleanliness of each sample. A one inch square was divided into ten segments on each side, yielding a 100 point grid. The grid was super-imposed on the 100X microstructures and each inclusion or second phase on an intercept point was counted. The counting process was repeated 10 times per sample for a total of 1000 points. A standard deviation calculation was included to show the significance of the scatter associated with the calculated volume fraction of inclusions. The inclusion volume fractions for chip and production ingots were compared to determine whether the use of chips as remelt scrap charge makeup increases inclusions in the resulting cast ingot.

Results and Discussion

The three chip heats and the control heat behaved like production heats in the VIR furnace during melting. Observation of the melt pool in the crucible showed a normal skin forming on the surface. Chip briquettes readily dissolved in the pool and no residual chip or briquette oxide skin or skull was noted on the melt pool surface. All four heats poured like production heats after the crucible was tapped.

Examination of the crucible after casting the heats showed the normal remaining skin of slag and metal. Chip remelt heats did not create more crucible slag than production remelt heats. Measured casting yields, shown in Table 7, were further evidence that chips did not signficantly affect the melting and casting process.

Ingot surfaces from each of these four heats were excellent. All four heats were processed through the VIR process as normal production heats and they behaved as such. Chips and chip briquettes did not have detrimental effects on VIR processing or physical ingot quality.

The results of normal production chemical analyses on all four heats are shown in Table 8. The control heat, along with the 5% and 15% briquette heats, passed M833 chemistry specifications. Titanium values (weight %) were nominal and the carbon values were on the desired lower end of the specification range. All other trace elements appeared in nominal quantities and looked good in these heats.

Heat 5245, the 20% chip briquette heat, did not meet M833 chemistry specifications. Its iron level, 56 ppm, exceeded the 50 ppm maximum value allowed under the M833 chemistry specification. The high iron concentration was traced to a high iron level (86 ppm) in one of the charge derbies.

heats had comparable microcleanliness with production heats. Tables 21 and 22 show the volume percent inclusions for experimental and production heats respectively. Figure 26 graphically demonstrates the relative inclusion levels for chip and production heats. While it would appear from Figure 26 that chip heats are slightly cleaner than production heats, this is deceptive since the standard deviation for the measurements is as large as the inclusion count in almost all cases; e.g., the amount of inclusions is at or near the limit of the measurement technique.

It was very important to determine that the inclusions which were present were the same in both production and chip ingots. Extensive studies were made on the microprobe to identify the inclusions. These studies showed that all the inclusions were titanium carbide. No oxide inclusions were found in either the production or experimental heats. Figures 27, 28, and 29 are secondary electron image Ti EDS and C WDS images of the second phase particles respectively. These figures conclusively show that the second phase is TiC.

Summary

Task B work has shown that machining chips can be added as briquettes to the VIR charge for large caliber core remelt heats in amounts up to 20 weight %. No adverse effects were experienced in the melting and casting process. Ingot chemistry was unaffected both with respect to nominal Ti and C levels and to trace element composition. Furthermore, the presence of recycle machining chips did not affect the longitudinal or radial elements segregation with the chip ranges studied. Lastly, the use of recycle machining chips did not create a microcleanliness problem in the cast ingots. No oxide inclusions were observed in the heats produced in Task B (none were found in the randomly selected production heats either).

TASK C - PRODUCTION HEAT WITH RECYCLED CHIPS

The studies conducted in Tasks A and B determined machining chip processing parameters and demonstrated that machining chips could be recycled in the VIR furnace and cast into acceptable M833 ingots. Accordingly, Task C was designed to prove out the use of recycled machining chips to produce specification M833 cores. One heat of DU-3/4 wt.%Ti was produced using recycle chips as part of the charge. The heat was processed as normal production material to produce finished cores with no special controls. Eighty of these cores were to be considered deliverables.

Procedure

A suggested procedure for processing the lathe chips to briquette form as feedstock for vacuum furnaces was submitted to and approved by AMCCOM. The procedure was based on the processes developed in Task A and verified in Task B. A copy of the chip processing procedure can be found in Appendix B.

A remelt charge containing 7% chip briquettes was used. This percentage was chosen because at this step of the evaluation, not knowing chip processing yields and recovery, it appeared to be a likely addition for recycle. Task B showed that remelt heats with chip briquettes were not sensitive to chip briquette quantities, at least in heats containing up to 20% chip briquettes. Both coarse and fine machining chips, in a ratio of 33% coarse to 67% fine, were collected and processed as shown in Figure 30.

A charge of 7% chips required 230 pounds of briquettes. Two derbies and process scrap also were used in the charge of this heat, #5287. All chips were collected and identified by the heat from which they were machined, Table 23. Heat 5287 was melted to normal Aerojet remelt procedures. The chip briquettes were loaded on the top of the other remelt charge in the crucible. See Table 24 for remelt charge makeup.

The billets were treated as an ordinary Aerojet production material and cores were produced following the M833 Process Control Document (PCD) for each process step. Figure 31 shows the overall processing for these cores. The billets were broken down to 1.30" diameter rod. Only 90 cores were finished, with the remainder of the heat left as OD turned blanks.

Discussion and Results

Heat 5287 melted the same as a typical production heat. Its pool looked normal as did the residual left in the crucible. There was no evidence of skulls on the melt pool surface or remaining in the crucible after casting.

Billet surface quality for heat 5287 was very good. All billet surfaces were quite smooth with no evidence of cold shuts or other rough surfaces. The yield was 85.5% which compared favorably with the heats in Task B. Chemical analysis showed the heat met all M833 chemistry specifications, Table 25.

Rod rolling to 1.3" diameter, blanking and solution heat treating and aging was routine. Mechanical properties were good and within M833 specification requirements, Table 26.

All bars from the heat were OD turned and submitted for ultrasonic testing. All bars passed the ultrasonic testing. All bars passed the surface hardness test.

Ninety bars were machined to finished cores. Eight bars were rejected for being out of tolerance during final machining. This is within the range encountered during production.

Heat 5287 confirmed that machining chips (briquettes) can be successfully utilized as feedstock for large caliber core heats with no detrimental effect on properties.

Process Modifications

At the completion of the effort to be performed under the guidelines of the statement of work, additional efforts were undertaken by AHMC personnel to future characterize and optimize the chip recycling process. Several process areas were evaluated, one being alternative pickling solutions, which is discussed in Appendix C. The alternative pickling was conducted as an amendment to the proposed statement of work (SOW) and performed as part of the government funded MM&T program.

Other specific areas investigated as potential process modifications were the elimination of the pickling and briquetting processing steps. These latter two efforts were conducted after all other work for the MM&T program, excluding the submittal of the final report. This work was beyond the basic or amended statement of work and, therefore, not funded by the MM&T program.

As previously stated, AHMC initiated, at the completion of all work performed for the basic and amended statement of work for this MM&T program, an investigation directed toward recycling machine turns and using these chips as make-up charge in production streams other than large caliber penetrator production. Although this work was performed by AHMC after the completion of the MM&T program and was totally Aerojet funded, the results of this post MM&T investigation identified a significant process optimization. The significance was such that it was felt that the results of these findings should be included as part of this report.

The findings eluded to in the above paragraph are the elimination of the pickling and briquetting chip recycle processing steps. Aerojet determined, through use of a simplified chip processing operation developed for a material stream which could withstand greater variations

in material properties, that pickling and briquetting of the chips was not required to obtain a high quality metal. Upon further analysis, it was concluded that eliminating pickling and briquetting from the chip processing operation imposed no detrimental effect to the resultant chemical or physical properties of the material when compared to current production.

To answer the question as to why pickling and briquetting were initially considered essential steps in the initial chip recycling process, the following information should be presented. When the proposal for establishing and proving out a process for uranium induction remelting of depleted uranium chips was written, there was considerable concern by both the government and Aerojet technical community over the cleanliness and handleability of DU machining chips. During the actual time period which this MM&T program was performed, the machining chips for this program were collected by use of individual chip conveyors at each lathe. The lack of sufficient coolant spray and dwell time allowed the chips to undergo a significant amount of oxidation. Since the completion of the actual work performed for this program, excluding submittal of the final report, AHMC has installed and implemented use of a central chip coolant system (CCCS). This unique machining chip handling system quickly cools the chips, greatly restricting the amount of oxidation which forms on the chip surface, thereby eliminating the need for pickling.

The benefits obtained from briquetting the processed chips were that compacted chips had a higher density than loose ones, allowing a greater proportion of chips to be loaded into the remelt furnace. The processed chips were found to oxidize very slowly once they had been cleaned and briquetting did not appear to affect this rate. When comparing the cost of equipment and labor necessary to briquette the cleaned chips to the benefits identified above, it was felt that briquetting was not cost effective and, therefore, removed as one of the basic processing steps.

Process cost savings alone does not totally justify the elimination of pickling as a processing step. The yields listed below for process machining chips from collection at the lathe through to drying shows that approximately a 15 percent increase in material savings can be obtained by eliminating pickling.

Chip Recycling Yields

Operation	Yield
Removal from Lathe	100%
CCCS	98%
Crushing	98%
Sieving	90%
Washing	99%
Pickling Operation	85%
Rinse	99%
Drying	99%

Therefore, for AHMC's unique situation, the pickling and briquetting processing steps appear to be economically unjustifiable and will not be included as part of Aerojet's process. As stated, this is unique to AHMC's facility and, therefore, these processing steps have been included in the subsequent sections.

ECONOMIC ANALYSIS

An economic analysis has been performed on recycling depleted uranium chips. This analysis indicates that over 200,000 lbs. of derby metal/per year would be saved assuming a production rate of 5000 M833 cores per month. The amount of manufacturing labor saved per year equals 200 hours as opposed to the additional labor required for the chip recycling process of 4000 hrs/year. Estimated recurring costs are approximately $50,000 per year.

The projected savings per core are listed below as are the additional equipment requirements of $622,000.

DU CHIP RECYCLING COST SAVINGS PER CORE

Based on the following assumptions:

- 1.32 lbs. UF_4 to produce 1 lb. DU metal (stoichiometric amount)
- 3.5 lbs. of usable chips saved per core
- 1.18 inch diameter rod stock
- Green salt costs approximately $2.00/lb.
- Magnesium (Mg) costs approximately $2.00/lb.
- 60 heats a year eliminated by recycling

TOTAL MATERIAL SAVINGS/CORE (UF_4, Mg, graphite, etc.)	$10.90
Disposal of Chips and Other Direct Waste/Core	$ 2.30
TOTAL SAVINGS/CORE	$13.10

Costs

Materials (Dies, Punches, Detergent, Acid, etc.)	
$\frac{\$50,000}{60,000 \text{ cores}}$	$ 0.83
Increased Direct Labor Costs	
$\frac{\$40,000}{60,000 \text{ cores}}$	$ 0.67
TOTAL COSTS/CORE	$ 1.50
TOTAL SAVINGS/CORE AFTER COSTS	$11.60/CORE
At 5000 cores/mo.	
TOTAL ANNUAL SAVING	$696,000

ADDITIONAL CHIP PROCESSING EQUIPMENT

Chip Collection, Inspection and Crushing		
Chip Transfer Conveyors Contaminant Inspection Station Crusher Upgrade		$ 48,000
Chip Sizing and Cleaning		
Automatic Screening System Washer Centrifugal Dryers Pickling System Chip Storage		$115,000
Chip Briquetting		
Fully Automated Press Briquette Storage Briquette Loader		$400,000
System Ventilation		$ 59,000
	TOTAL	$622,000

This chip recycling system would be capable of satisfying both normal and mobilization production rates on a 3/8/5 basis.

The minimum equipment to produce 10,000 penetrators per month would cost $242,000. A breakdown of the equipment cost is listed below. The major difference between the recommended facilitization costs and the minimum costs involve the briquetting press. The recommended facilitization effort utilizes an automated press allowing all briquetting on one shift. The minimum facilitization effort requires three shifts to complete the briquetting operation, with three times the labor hours. This increased labor will not affect materially the cost/core since it was based on use of non-automated briquetting press, e.g. they represent a worst case cost situation.

MINIMAL CHIP PROCESSING EQUIPMENT

Chip Collection, Inspection and Crushing		
Chip Transfer Conveyors Containment Inspection Status Crusher Upgrade		$ 30,000
Chip Sizing and Cleaning		
Automatic Screening System Washer Pickling System Chip Storage		$103,000
Chip Briquetting		
Upgrade Press Briquette Storage Briquette Loader		$ 50,000
System Ventilation		$ 59,000
	TOTAL	$242,000

HEALTH AND SAFETY HAZARDS EVALUATION

There were several Health and Safety questions relative to future implementation of chip recycling which were studied. Health issues studied included the air quality in the immediate vicinity of the equipment. We also were concerned with potential water quality problems with respect to the ionic detergent wash and the nitric acid pickling. Safety concerns centered around the potential for fires at the ring crusher and how they would be eliminated.

Health

Air Quality - Air quality was very good around all the processing equipment including the ring crusher. Standard DU manufacturing ventilation procedures and equipment were used at those operations where chips were crushed and/or pickled. These operations were monitored using industry standard air sampling techniques and no readings higher than 15% of acceptable limits were found. The highest reading around the ring crusher was 15% of the acceptable limit. The normal operating procedure is to shut down equipment or require respirators for the operators if the air particulate level reaches 25% of the acceptable limit.

Nitrous oxide emissions were a potential hazard if the chips were pickled at elevated temperatures. The rapid emission of nitrous oxide could overload the scrubber and cause visible air quality problems. This problem was solved by pickling at lower temperatures where the slower pickling kinetics with the resulting lower emission of nitrous oxide did not exceed the scrubber's capacity.

Water Quality - Two water quality problems are anticipated if chip recycling enters production. Both the ionic detergent chip washing and the nitric acid pickling solutions present problems for waste water treatment.

The ionic detergent, which removed the coolant lubricant, presented a disposal problem similar to the coolant itself. The use of this detergent will require the same special disposal means required for the coolant.

A second water quality problem will be the effect of increased nitric acid pickling on nitrate emissions. The increased nitric acid pickling usage associated with production chip recycling may push nitrate emissions close to the maximum allowable limit of 100 lbs. per day. A study is being pursued to determine if an alternative pickling system with easily managed waste products can replace some or all of the nitric acid pickling.

Safety

The over-riding safety concern was associated with the fires in the ring crusher during Task A. The chips were being crushed dry and collected in a 20 gallon barrel located directly beneath the crusher output. Several fires were caused by loose isolated chips sparking during the crushing and finally igniting. Filling the collector two-thirds full with coolant significantly reduced the possibility of fire since the chips were diluged and smothered with coolant.

Even after this modification, two fires occurred after use of the coolant was implemented. It was determined that stray chips were missing the barrel and the coolant and igniting. A ducting system was constructed which acted as a funnel between the ring crusher assembly and the collection barrel. All chips were guided into the barrel. The combination of the coolant and the ducting system eliminated fires in the remainder of Task A and throughout the processing of about 2500 lbs. of chips in Task B and C.

From this work, it was concluded that if chip recycling were implemented all chips should be carefully contained in a cool environment, particularly during potential heat generating processes. For example, a conveyor for chips should have a chute of coolant beneath it to catch and cool any stray chips. This would be particularly important near a shearing or crushing operation.

One important aspect of crushing is the removal of small foreign material such as tungsten carbide inserts. Inserts in the spinning ring crusher could produce significant sparking with a much greater chance of fire. A pre-crushing inspection is a very good preventative means of eliminating this added fire hazard.

Another simple means to eliminate fires in the ring crusher is to recirculate coolant through the crusher. Coolant would smother sparks before a fire had a chance to start. The current ring crusher could be easily modified to recirculate coolant and almost certainly eliminate the potential of fire.

Except for crushing, chip processing would follow all the normal safety considerations which are used throughout the plant. Chip recycling can be a safe operation in production with proper design and operating precautions.

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of this program, Aerojet has concluded that staballoy (depleted uranium - .75% wt. titanium) machining chips can be successfully processed as vacuum induction remelting scrap charge material. Furthermore, it was proven that specification M833 cores can be produced from a heat of material containing 7% recycled DU-.75% wt. Ti machining chips.

Finally, it was concluded that the process developed was shown to be economically attractive.

Aerojet recommends that serious consideration be given to implementing the use of recycled machining chips for production of large caliber cores. Furthermore, it is recommended that an investigation for alternative cleaning methods be conducted to eliminate the need for nitric acid pickling.

TABLE 1

CHIP BLENDS STUDIED IN TASK A

Group Number	Group Constituents (%)	Chip Type
1	100 OD Chips	Coarse
2	33 Finishing Chips 67 OD Chips	Fine Coarse
3	50 Finishing Chips 50 OD Chips	Fine Coarse
4	84 Finishing Chips 16 OD Chips	Fine Coarse
5	100 Finishing Chips	Fine

TABLE 2

ACID PICKLING TRIALS

TRIAL NUMBER	CHIP TYPE (%)	ACID TEMPERATURE	ACID CONCENTRATION	IMMERSION TIME	CHIP APPEARANCE AFTER PICKLING	COMMENTS
1.	100 OD	115°F (46°C)	5.2 N (Production Acid)	45 seconds	Dark golden/ bluish	Excessive NO_x emissions, evacuated area. Insufficient oxide removal.
2.	100 OD	65°F (18°C)	2.0 N (Dilute)	8 minutes	Dark golden	No visible NO_x emissions. Insufficient oxide removal.
3.	100 OD	71°F (22°C)	4.3 N (Dilute)	10 minutes	Light golden	No visible NO_x emissions. Good oxide removal.
4.	100 Final	73°F (23°C)	7.0 N (Production Acid)	6 minutes	Light golden	Some visible NO_x emissions, contained within ventilation enclosure. Good oxide removal.
5.	84 Final 16 OD	81°F (27°C)	5.6 N (Production Acid)	9 minutes	Light golden	Some visible NO_x emissions, contained within ventilation enclosure. Good oxide removal.
6.	33 Final 67 OD	84°F (29°C)	7.9 N (Production Acid)	12 minutes	Light golden	Some visible NO_x emissions, contained within ventilation enclosure. Good oxide removal.
7.	50 Final 50 OD	85°F (29°C)	7.9 N (Production Acid - 4/12 concen.)	12 minutes	Light golden	Some visible NO_x emissions, contained within ventilation enclosure. Good oxide removal.

TABLE 3

BULK YIELDS THROUGH PROCESSING

TEST GROUP NUMBER	GROUP CONSTITUENTS (%)	LOSSES TO SCREENING (%)	LOSSES TO PICKLING (%)	NET YIELD (%)
1	100 OD CHIPS	3.8	10.7	85.9
2	33 FINISHING/ 67 OD	19.3	10.7	72.0
3	50 FINISHING/ 50 OD	24.7	14.2	64.7
4	84 FINISHING/ 16 OD	19.2	12.5	70.7
5	100 FINISHING	19.1	18.1	66.3

TABLE 4

CHEMICAL ANALYSIS OF CHIPS

	Parts Per Million						Percentage	
	Fe	Ni	Si	C	Pb	H_2	Ti	O_2
100% OD CHIPS (Heat 4727)								
Baseline heat chemistry	22	8	38	35	--	--	0.76	--
Unprocessed chips	22	11	43	35	11	--	0.77	--
After crushing	24	12	47	39	11	--	0.75	--
After wash & pickle	25	10	46	40	11	0.8	0.75	0.044 0.029
100% FINISHING CHIPS (Heat 4711)								
Baseline heat chemistry	17	22	52	34	--	--	0.75	--
Unprocessed chips	17	11	40	43	8	--	0.77	--
After crushing	20	11	37	59	8	--	0.77	--
After wash & pickle	17	10	28	41	8	2.1	0.77	0.060 0.079

TABLE 5

PHYSICAL ANALYSIS OF BRIQUETTES

TEST GROUP	BRIQUETTE GROUP (%)	BRIQUETTES	DENSITY	% THEORETICAL DENSITY	SURFACE	COLOR	SURFACE CONDITION
1	100 OD CHIPS	6	0.2763	40.9	COARSE	LIGHT GOLDEN	GOOD, SOME SPALLING FROM TOP & BOTTOM SURFACES
2	33 FINISH/ 67 OD	6	0.2922	43.3	SMOOTH	MEDIUM GOLDEN	EXCELLENT
3	50 FINISH 50 OD	6	0.2948	43.7	SMOOTH	MEDIUM GOLDEN	EXCELLENT
4	84 FINISHING/ 16 OD	6	0.3023	44.8	SMOOTH	MEDIUM GOLDEN	EXCELLENT
5	100 FINISHING CHIPS	6	0.3009	44.6	SMOOTH	MEDIUM GOLDEN	EXCELLENT

TABLE 6

CHIP BRIQUETTE REMELT CHARGE MAKEUP

Heat No.	% Derby	% Process Scrap	Weight Chips,* lbs.	% Chips*	Total Charge (lbs.)
5056	60.7	39.3	-0-	-0-	3400
5072	60.7	35.8	168	5.0	3400
5130	62.0	22.8	509	15.2	3350
5245	60.5	19.7	664	19.8	3350

*as pressed briquettes

TABLE 7

MELTING AND CASTING YIELDS FOR TASK B & C HEATS

Heat Number	Net Yield
5056*	85.7%
5072**	82.8%
5130**	83.2%
5245**	81.8%
5287***	85.5%

*No chips heat (control - not production)
**Recycle chip heat
***Pilot production recycle chip heat

TABLE 8

CHEMISTRY FROM ANALYTICAL SAMPLING
ppm (Except for Titanium)

Heat No.	Al	Cd	Co	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti*	V	Zn	C
5056	3.0	0.3	1.0	7.8	5.4	15	2.5	8.0	8.0	47	.74	1.1	7.1	36.6
5072	3.0	0.3	0.8	4.7	5.4	15	2.5	6.8	14	41	.74	1.1	7.1	31.1
5130	8.3	0.3	1.0	4.7	5.4	20	2.5	6.8	6.8	45	.74	1.1	7.1	27.6
5245	8.3	0.3	1.0	7.8	5.4	R 56	2.5	6.8	12	24	.72	1.1	7.1	44.2

*wt. %

R - Does not meet TDP - Reject

TABLE 9

DETAILED INGOT CHEMISTRY PROFILE OF HEAT 5056 - CONTROL HEAT

Sample #***	Al	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti	C	Pb
11	<8.3*	<7.8*	<5.4*	22	<2.5*	<6.8*	6.8	50	.76	32	16
12	<8.3	<7.8	<5.4	25	<2.5	<6.8	6.8	54	.77	29	13
13	<8.3	<7.8	<5.4	26	<2.5	<6.8	6.8	50	.74	45	15
21	<8.3	<7.8	<5.4	19	<2.5	<6.8	<6.8*	45	.75	32	10.8
22	<8.3	<7.8	<5.4	20	<2.5	<6.8	<6.8	40	.76	47	15
23	<8.3	<7.8	<5.4	25	<2.5	<6.8	<6.8	50	.76	23	15
31	<8.3	<7.8	<5.4	20	<2.5	<6.8	<6.8	40	.77	33	13
32	<8.3	<7.8	<5.4	25	<2.5	<6.8	<6.8	35	.74	22	14
33	<8.3	<7.8	<5.4	30	<2.5	<6.8	<6.8	35	.75	30	12
41	<8.3	<7.8	<5.4	25	<2.5	<6.8	<6.8	40	.73	55	9.0
42	<8.3	<7.8	<5.4	25	<2.5	<6.8	<6.8	40	.77	26	10
43	<8.3	<7.8	<5.4	20	<2.5	<6.8	<6.8	40	.77	31	10
51	<8.3	<7.8	<5.4	19	<2.5	<6.8	<6.8	40	.77	30	10.8
52	<8.3	<7.8	4**	<19*	<2.5	<6.8	9	19	.77	26	<5.8*
53	<8.3	<7.8	4	<19	<2.5	<6.8	8	40	.76	32	<5.8
61	<8.3	<7.8	5.4	20	<2.5	<6.8	9	43	.74	29	5.8
62	<8.3	<7.8	4	20	<2.5	<6.8	9	34	.74	38	5.8
63	<8.3	<7.8	4	<19	<2.5	<6.8	9	18	.74	32	5.8
71	<8.3	<7.8	<5.4	19	<2.5	<6.8	8.0	24	.75	28	<5.8
72	<8.3	<7.8	<5.4	19	<2.5	<6.8	8.0	18	.75	27	<5.8
73	<8.3	<7.8	<5.4	20	<2.5	<6.8	8.0	24	.77	24	<5.8
81	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	24	.76	26	<5.8
82	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	20	.77	25	<5.8
83	<8.3	<7.8	<5.4	19	<2.5	<6.8	8.0	24	.78	23	<5.8
91	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	20	.76	30	<5.8
92	<8.3	<7.8	<5.4	19	<2.5	<6.8	8.0	20	.77	30	<5.8
93	<8.3	<7.8	<5.4	20	<2.5	<6.8	8.0	20	.77	29	<5.8
101	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	20	.76	28	<5.8
102	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	20	.78	29	<5.8
103	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	24	.75	28	<5.8
111	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	20	.76	21	<5.8
112	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	30	.78	29	<5.8
113	<8.3	<7.8	<5.4	<19	<2.5	<6.8	8.0	20	.76	29	<5.8
121	<8.3	<7.8	<5.4	21	<2.5	<6.8	8.0	44	.71	30	<5.8
122	<8.3	<7.8	<5.4	20	<2.5	<6.8	8.0	30	.72	43	<5.8
123	<8.3	<7.8	<5.4	19	<2.5	<6.8	8.0	24	.70	27	<5.8

*Threshold detectable limit
**Different calibration curve accounts for the lower threshold value
***Sample # Code

XX — Slice Location

X — Location Within Slice

Slice Location
1 - Top of Billet
12 - Toe of Billet

Location Within Slice
1. O.D.
2. Mid-Radius
3. Centerline

TABLE 10

DETAILED INGOT CHEMISTRY PROFILE OF HEAT 5072 - 5% CHIP BRIQUETTE HEAT

Sample #	Al	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti	C	Pb
11	<8.3*	<7.8*	<5.4*	20	<2.5*	<6.8*	12	56	.76	17	5.8
12	<8.3	<7.8	<5.4	<19*	<2.5	<6.8	<12	34	.79	23	<5.8*
13	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	54	.78	29	<5.8
21	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	48	.79	23	<5.8
22	<8.3	<7.8	<5.4	<19	<2.5	<6.8	12	50	.78	24	5.8
23	<8.3	<7.8	<5.4	<19	<2.5	<6.8	12	52	.76	23	5.8
31	<8.3	<7.8	<5.4	<19	<2.5	<6.8	12	50	.78	21	6
32	<8.3	<7.8	<5.4	<19	<2.5	<6.8	15	66	.76	18	6
33	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	26	.77	21	5.8
41	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	38	.76	22	5.8
42	<8.3	<7.8	<5.4	<19	<2.5	<6.8	13	52	.76	18	5.8
43	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	42	.79	23	<5.8
51	<8.3	<7.8	<5.4	19	<2.5	<6.8	12	46	.78	22	<5.8
52	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	44	.77	21	<5.8
53	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	48	.78	36	5.8
61	<8.3	<7.8	5.4	<19	<2.5	<6.8	13	50	.77	25	5.8
62	<8.3	<7.8	<5.4	<19	<2.5	<6.8	12	46	.78	23	5.8
63	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	30	.79	30	<5.8
71	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	44	.75	24	<5.8
72	<8.3	<7.8	<5.4	<19	<2.5	<6.8	13	50	.77	23	<5.8
73	<8.3	<7.8	<5.4	<19	<2.5	<6.8	13	54	.79	22	<5.8
81	<8.3	<7.8	<5.4	<19	<2.5	<6.8	13	34	.79	24	<5.8
82	<8.3	<7.8	<5.4	<19	<2.5	<6.8	13	54	.77	22	<5.8
83	<8.3	<7.8	<5.4	<19	<2.5	<6.8	12	50	.77	23	<5.8
91	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	43	.77	24	<5.8
92	<8.3	<7.8	<5.4	<19	<2.5	<6.8	12	45	.76	22	<5.8
93	<8.3	<7.8	<5.4	<19	<2.5	<6.8	13	48	.79	19	<5.8
101	<8.3	<7.8	<5.4	28	<2.5	<6.8	17	56	.77	22	<5.8
102	<8.3	<7.8	<5.4	<19	<2.5	<6.8	<12	53	.75	25	<5.8
103	<8.3	<7.8	<5.4	<19	<2.5	<6.8	12	53	.77	24	<5.8
111	<8.3	<7.8	<5.4	26	<2.5	<6.8	12	51	.77	25	<5.8
112	<8.3	<7.8	<5.4	33	<2.5	<6.8	12	54	.77	23	<5.8
113	<8.3	<7.8	<5.4	22	<2.5	<6.8	12	51	.76	21	<5.8
121	<8.3	<7.8	<5.4	26	<2.5	<6.8	12	48	.77	20	<5.8
122	<8.3	<7.8	<5.4	29	<2.5	<6.8	18	54	.77	25	<5.8
123	<8.3	<7.8	<5.4	22	<2.5	<6.8	12	48	.76	23	<5.8

*Threshold detectable limit

TABLE 11

DETAILED INGOT CHEMISTRY PROFILE OF HEAT 5130 - 15% CHIP BRIQUETTE HEAT

Sample #	Al	H_2	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti	C	Pb
11	<8.3	3.3	<7.8*	9	20	<2.5*	<6.8	9	53	.72	21	<5.8*
12	<8.3	3.2	<7.8	9	21	<2.5	<6.8	10	56	.76	20	<5.8
13	<8.3		<7.8	5.4	20	<2.5	<6.8	9	50	.75	27	<5.8
21	<8.3		<7.8	5.4	20	<2.5	<6.8	9	40	.76	24	<5.8
22	<8.3		<7.8	5.4	19*	<2.5	<6.8	9	50	.76	26	<5.8
23	<8.3	4.2	<7.8	4	<19	<2.5	<6.8	9	37	.76	24	<5.8
31	<8.3		<7.8	5.4	20	<2.5	<6.8	9	43	.77	24	<5.8
32	<8.3		<7.8	5.4	20	<2.5	<6.8	9	46	.74	32	<5.8
33	<8.3	3.3	<7.8	5.4	19	<2.5	<6.8	9	34	.75	30	<5.8
41	<8.3		<7.8	5.4	19	<2.5	<6.8	8	22	.75	24	<5.8
42	<8.3		<7.8	5.4	<19	<2.5	<6.8	8	34	.77	20	<5.8
43	<8.3	1.2	<7.8	5.4	19	<2.5	<6.8	8	17	.74	26	<5.8
51	<8.3		<7.8	4	20	<2.5	<6.8	9	53	.76	21	<5.8
52	<8.3	2.9	<7.8	<5.4*	23	<2.5	<6.8	7.1	40	.75	25	17
53	<8.3	3.2	<7.8	<5.4	24	<2.5	<6.8	9	50	.74	27	23
61	<8.3		<7.8	<5.4	23	<2.5	<6.8	<6.8*	38	.75	27	18
62	<8.3	2.7	<7.8	<5.4	23	<2.5	<6.8	7.1	48	.76	29	21
63	<8.3		<7.8	<5.4	23	<2.5	<6.8	6.8	42	.77	25	18
71	<8.3		<7.8	<5.4	23	<2.5	<6.8	<6.8	35	.74	25	18
72	<8.3		<7.8	<5.4	22	<2.5	<6.8	<6.8	35	.74	24	14
73	<8.3		<7.8	<5.4	23	<2.5	<6.8	<6.8	43	.76	31	16
81	<8.3		<7.8	<5.4	23	<2.5	<6.8	7.1	42	.74	27	18
82	<8.3		<7.8	<5.4	24	<2.5	<6.8	8	49	.78	24	22
83	<8.3	2.7	<7.8	<5.4	22	<2.5	<6.8	7	47	.78	20	20
91	<8.3		<7.8	<5.4	23	<2.5	<6.8	7.1	46	.74	19	17
92	<8.3		<7.8	<5.4	21	<2.5	<6.8	<6.8	44	.76	18	19
93	<8.3		<7.8	<5.4	25	<2.5	<6.8	6.8	50	.77	27	19
101	<8.3		<7.8	<5.4	20	<2.5	<6.8	9.0	44	.76	22	17
102	<8.3		<7.8	<5.4	21	<2.5	<6.8	6.8	44	.76	21	19
103	<8.3		<7.8	<5.4	22	<2.5	<6.8	7.0	45	.79	27	16
111	<8.3		<7.8	<5.4	30	<2.5	<6.8	7.0	50	.77	37	22
112	<8.3		<7.8	<5.4	25	<2.5	<6.8	6.8	40	.78	25	17
113	<8.3		<7.8	<5.4	23	<2.5	<6.8	8.0	50	.79	22	17
121	<8.3		<7.8	<5.4	21	<2.5	<6.8	7.0	43	.76	26	16
122	<8.3		<7.8	<5.4	22	<2.5	<6.8	6.8	44	.76	23	16
123	<8.3		<7.8	<5.4	24	<2.5	<6.8	8.0	44	.76	18	18

*threshold detectable limit

**due to the sampling technique, some ingot hydrogen data show a significant scatter and, therefore, was not included.

TABLE 12

DETAILED INGOT CHEMISTRY PROFILE OF HEAT 5245 - 20% CHIP BRIQUETTE HEAT

Sample #	Al	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti	C	Pb
11	<8.3*	<7.8*	<5.4*	>111	<2.5*	<6.8*	21	67	.73	37	<5.8*
12	<8.3	<7.8	<5.4	>111	<2.5	<6.8	21	53	.73	44	<5.8
13	<8.3	<7.8	<5.4	>111	<2.5	<6.8	21	56	.71	39	<5.8
21	<8.3	<7.8	<5.4	>111	<2.5	<6.8	21	54	.74	40	<5.8
22	<8.3	<7.8	<5.4	>111	<2.5	14	16	49	.74	33	<5.8
23	<8.3	<7.8	<5.4	103	<2.5	22	18	51	.72	32	<5.8
31	<8.3	<7.8	<5.4	81	<2.5	16	13	43	.72	35	<5.8
32	<8.3	<7.8	<5.4	81	<2.5	16	13	47	.75	40	<5.8
33	<8.3	<7.8	<5.4	66	<2.5	16	16	38	.70	28	<5.8
41	<8.3	<7.8	<5.4	111	<2.5	17	17	48	.72	36	<5.8
42	<8.3	<7.8	<5.4	95	<2.5	14	16	47	.72	35	<5.8
43	<8.3	<7.8	<5.4	96	<2.5	12	12	41	.74	38	<5.8
51	<8.3	<7.8	<5.4	50	<2.5	13	15	45	.74	42	<5.8
52	<8.3	<7.8	<5.4	111	<2.5	17	15	54	.72	35	<5.8
53	<8.3	<7.8	<5.4	111	<2.5	20	15	51	.75	41	<5.8
61	<8.3	<7.8	<5.4	96	<2.5	16	14	43	.73	45	<5.8
62	<8.3	<7.8	<5.4	56	<2.5	12	12	37	.74	34	<5.8
63	<8.3	<7.8	<5.4	56	<2.5	17	13	35	.74	40	<5.8
71	<8.3	<7.8	<5.4	58	<2.5	14	12	32	.75	48	<5.8
72	<8.3	<7.8	<5.4	55	<2.5	14	13	32	.73	39	<5.8
73	<8.3	<7.8	<5.4	48	<2.5	12	<12	27	.75	40	<5.8
81	<8.3	<7.8	<5.4	55	<2.5	12	13	32	.74	41	<5.8
82	<8.3	<7.8	<5.4	>111	<2.5	<6.8	16	41	.73	40	<5.8
83	<8.3	<7.8	<5.4	>111	<2.5	<6.8	16	44	.75	44	<5.8
91	<8.3	<7.8	<5.4	111	<2.5	<6.8	15	38	.74	46	<5.8
92	<8.3	<7.8	<5.4	96	<2.5	<6.8	16	39	.74	41	<5.8
93	<8.3	<7.8	<5.4	>111	<2.5	<6.8	17	38	.75	46	<5.8
101	<8.3	<7.8	<5.4	>111	<2.5	<6.8	16	44	.74	39	<5.8
102	<8.3	<7.8	<5.4	111	<2.5	<6.8	15	43	.73	42	<5.8
103	<8.3	<7.8	<5.4	>111	<2.5	<6.8	16	37	.74	41	<5.8
111	<8.3	<7.8	<5.4	>111	<2.5	<6.8	15	41	.75	35	<5.8
112	<8.3	<7.8	<5.4	111	<2.5	<6.8	15	42	.76	36	<5.8
113	<8.3	<7.8	<5.4	>111	<2.5	<6.8	16	39	.76	28	<5.8
121	<8.3	<7.8	<5.4	81	<2.5	<6.8	13	38	.77	49	<5.8
122	<8.3	<7.8	<5.4	>111	<2.5	<6.8	15	36	.76	44	<5.8
123	<8.3	<7.8	<5.4	88	<2.5	<6.8	16	37	.77	45	<5.8

*threshold detectable limit

TABLE 13

RADIAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEAT
MEAN+ $\hat{\sigma}_{n-1}$ BY LOCATION ON SLICES

Heat 5056A	Fe		Si		C		Pb	
Slice	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$
Edge	22.6	8.5	33.8	11.4	31.2	8.2	8.4	3.5
Mid-Radius	20.8	2.6	30.0	11.2	30.9	7.6	8.3	3.8
Center	21.2	3.7	30.8	11.7	29.4	5.9	8.3	3.8

TABLE 14

RADIAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEAT
MEAN+ $\hat{\sigma}_{n-1}$ BY LOCATION ON SLICES

Heat 5072A	Fe		Si		C		Pb	
Slice	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$
Edge	21.0	3.5	47.0	6.6	22.4	2.3	5.8	0.06
Mid-Radius	21.0	4.7	50.2	7.7	22.2	2.3	5.8	0.06
Center	19.5	1.2	46.3	9.2	24.5	4.8	5.8	0

TABLE 15

RADIAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEAT
MEAN+ $\hat{\sigma}_{n-1}$ BY LOCATION ON SLICES

Heat 5130A	Fe		Si		C		Pb	
Slice	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$
Edge	21.8	3.0	42.4	8.5	24.8	4.6	14.0	6.4
Mid-Radius	21.7	1.9	44.2	6.3	23.9	4.0	13.9	6.4
Center	21.9	2.2	42.4	9.6	25.8	3.2	14.2	6.5

TABLE 16

RADIAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEAT
MEAN+ On BY LOCATION ON SLICES

Heat 5245A	Fe		Si		C		Pb	
Slice	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$	Mean	$\hat{\sigma}_{n-1}$
Edge	90.6	24.6	43.8	9.6	41.1	5.0	5.8	0
Mid-Radius	96.8	21.5	43.3	6.8	38.6	3.9	5.8	0
Center	93.6	23.7	41.2	8.1	38.5	6.1	5.8	0

TABLE 17

LONGITUDINAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEATS

Heat 5056	Fe		Si		C		Pb		Ti (Wt. %)	
Slice*	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}
1	24.3	2.1	51.3	2.3	35.3	8.5	14.7	1.5	.757	.015
2	21.3	3.2	45	5	34	12.1	13.6	2.4	.757	.006
3	25	5	36.7	2.9	28.3	5.7	13	1	.753	.015
4	23.3	2.9	40	0	37.3	15.5	10.2	1.0	.757	.023
5	19	0	31.3	10.9	29.3	3.0	7.5	2.9	.763	.006
6	19.7	0.58	31.7	12.7	33	4.6	5.8	0	.74	0
7	19.3	0.58	22	3.5	26.3	2.1	5.8	0	.757	.012
8	19	0	22.7	2.3	24.7	1.5	5.8	0	.77	.01
9	19.3	0.58	20	0	29.7	0.58	5.8	0	.767	.006
10	19	0	21.3	2.3	28.3	0.58	5.8	0	.763	.015
11	19	0	23.3	5.8	21.3	2.3	5.8	0	.767	.01
12	20	1	32.7	10.3	33.3	8.5	5.8	0	.71	.01

σ_{n-1} is the standard deviation for sampled data.

TABLE 18

LONGITUDINAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEATS

Heat 5072	Fe		Si		C		Pb		Ti (Wt. %)	
Slice*	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}
1	19.3	0.47	48	9.9	23	4.9	5.8	0	.777	.015
2	19.0	0*	50	2.0	23.3	0.6	5.8	0	.777	.015
3	19.0	0	47.3	20.1	20	1.7	5.9	0.11	.77	.01
4	19.0	0	44	7.2	21	2.6	5.8	0	.77	.017
5	19.0	0	46	2	26.3	8.4	5.8	0	.777	.006
6	19.0	0	42	10.6	26	3.6	5.8	0	.78	.01
7	19.0	0	49.3	5.0	23	1.0	5.8	0	.77	.02
8	19.0	0	46	10.6	23	1.0	5.8	0	.777	.012
9	19.0	0	45.3	2.5	21.7	2.5	5.8	0	.77	.015
10	22	5.2	54	1.7	23.7	1.5	5.8	0	.763	.012
11	27	5.6	52	1.7	23	2.0	5.8	0	.777	.006
12	25.7	3.5	50	3.5	27.7	2.5	5.8	0	.767	.006

*Pertaining to Tables 17-20: #1 slice is from top of billet; #12 is from toe.

TABLE 19

LONGITUDINAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEATS

Heat 5130	Fe		Si		C		Pb		Ti (Wt. %)	
Slice*	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}
1	20.3	0.58	53	3	22.7	3.8	5.8	0	.743	.021
2	19.3	0.58	42.3	6.8	24.7	1.2	5.8	0	.76	0
3	19.7	0.58	41	6.2	28.7	4.2	5.8	0	.753	.015
4	19	0	24.3	8.7	23.3	3.0	5.8	0	.753	.015
5	22.3	2.1	47.7	6.8	24.3	3.0	15.3	8.7	.75	.01
6	23	0	42.7	5.0	27.0	2.0	19	1.7	.76	.01
7	22.7	0.58	37.7	4.6	26.7	3.8	16	2	.747	.012
8	23	1	46	3.6	23.7	3.5	20	2	.767	.023
9	23	2	46.7	3.0	21.3	4.9	18.3	1.2	.757	.015
10	21	1	44.3	0.58	23.3	3.2	17.3	1.5	.77	.017
11	26	3.6	46.7	5.8	28	7.9	18.7	2.9	.78	.01
12	22.3	1.5	43.7	0.58	22.3	4.0	16.7	1.2	.76	0

Slice 1 - Top of Billet

↓

Slice 12 - Toe of Billet

TABLE 20

LONGITUDINAL CHEMICAL ANALYSIS FOR CHIP BRIQUETTE HEATS

Heat 5245	Fe		Si		C		Pb		Ti (Wt. %)	
Slice*	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}	Mean	σ_{n-1}
1	111	0	58.7	7.4	40	3.6	5.8	0	.723	.011
2	108.3	4.6	51.3	2.5	35	4.4	5.8	0	.733	.012
3	76	8.7	42.7	4.5	34.3	6.0	5.8	0	.723	.025
4	101	8.7	45.3	3.8	36.3	1.5	5.8	0	.727	.012
5	90.7	35.2	50.0	4.6	39.3	3.8	5.8	0	.737	.015
6	69.3	23.1	38.3	4.1	39.7	5.5	5.8	0	.737	.006
7	53.7	5.1	30.3	2.9	42.3	4.9	5.8	0	.743	.012
8	92.3	32.3	39.0	6.2	41.7	2.1	5.8	0	.740	.01
9	106	8.7	38.3	0.57	44.3	2.9	5.8	0	.743	.006
10	111	0	41.3	3.8	40.7	1.5	5.8	0	.737	.006
11	111	0	40.7	1.5	33	4.3	5.8	0	.757	.006
12	93.3	15.7	37.0	1	46	2.6	5.8	0	.767	.006

*Pertaining to Tables 17-20: #1 slice is from top of billet; #12 is from toe.

TABLE 21

EXPERIMENTAL BILLET MICROCLEANLINESS RATING

Heat and Billet Number	Billet Location	Microstructure Location*	Inclusion Count	Inclusion Count Standard Deviation	Percentage Inclusions
5056A	Top	O	.005	.005	0.5
		M	.008	.009	0.8
		C	.105	.315	1.05
5056A	Bottom	O	.012	.009	1.2
		M	.005	.007	0.5
		C	.008	.009	0.8
5072A	Top	O	.007	.008	0.7
		M**			
		C	.01	.008	1.0
5072A	Bottom	O	.005	.008	0.5
		M	.006	.007	0.6
		C	.011	.012	1.1
5130A	Top	O**			
		M	.008	.008	0.8
		C	.004	.007	0.4
	Bottom	O	.005	.007	0.5
		M	.008	.010	0.8
		C	.008	.012	0.8
5245A	Top	O	.007	.007	0.7
		M	.008	.006	0.8
		C	.006	.008	0.6
	Bottom	O	.009	.013	0.9
		M	.009	.009	0.9
		C	.017	.013	1.7

*O = O.D.; M = Mid-radius; C = Centerline

**Information excluded due to erroneous data

TABLE 22

PRODUCTION BILLET MICROCLEANLINESS RATING

Heat and Billet Number	Billet Location	Microstructure Location*	Inclusion Count	Inclusion Count Standard Deviation	Percentage Inclusions
5251AA	Top	O	.013	.013	1.3
		M	.012	.011	1.2
		C	.011	.009	1.1
	Bottom	O	.018	.010	1.8
		M	.013	.007	1.3
		C	.014	.008	1.4
5256LA	Top	O	.016	.013	1.6
		M	.015	.011	1.5
		C	.011	.011	1.1
	Bottom	O	.021	.010	2.1
		M	.020	.011	2.0
		C	.012	.009	1.2
5240GA	Top	O	.005	.007	0.5
		M	.006	.007	0.6
		C	.007	.008	0.7
	Bottom	O	.015	.014	1.5
		M	.011	.007	1.1
		C	.011	.009	1.1
5255LA	Top	O	.012	.012	1.2
		M	.017	.018	1.7
		C	.006	.008	0.6
	Bottom	O	.017	.012	1.7
		M	.020	.022	2.0
		C	.022	.014	2.2
5253LA	Top	O	.014	.010	1.4
		M	.017	.007	1.7
		C	.012	.008	1.2
	Bottom	O	.014	.015	1.4
		M	.011	.011	1.1
		C	.018	.014	1.8

*O = O.D.; M = Mid-radius; C = Centerline

TABLE 23

LATHE TURNINGS FOR 7% CHIP BRIQUETTE HEATS

	Heat No.
Barrel #3	5113
	5158
Barrel #9	5113
	5281
	5158
Barrel #1	5113
	5158

TABLE 24

CHIP BRIQUETTE HEAT 5287 REMELT CHARGE MAKEUP

% Derby	% Process Scrap	Briquettes	% Briquettes	Total Charge Weight
61.2 (2050 lbs)	31.9 (1068 lbs)	230	6.9	3350

TABLE 25

HEAT 5287 CHEMICAL PROPERTIES

Sample #	Cu	Fe	Ni	Si	Ti	C
AB	5.4	21	10	44	0.78	33
HT					0.76	28
BB					0.76	42
BT					0.77	25
CB					0.79	37
CT					0.78	31
DB					0.79	43
DT					0.77	48
EB					0.78	37
ET					0.78	22
GB					0.78	37
GT					0.75	20
HB					0.78	38
HT					0.79	37
JB					0.78	38
JT					0.75	25
KB					0.78	29
KT					0.77	24
LB					0.77	39
LT					0.77	33

TABLE 26

RECYCLE CHIP HEAT #5287
MECHANICAL PROPERTIES

Tensile

Ult. (ksi)	214
Yield (ksi)	141
% Elongation	24.3
% RA	21.0

Charpy

K_Q (ksi$\sqrt{in}$)	35.5

Profile Hardness (Avg.)

Rc	43.2

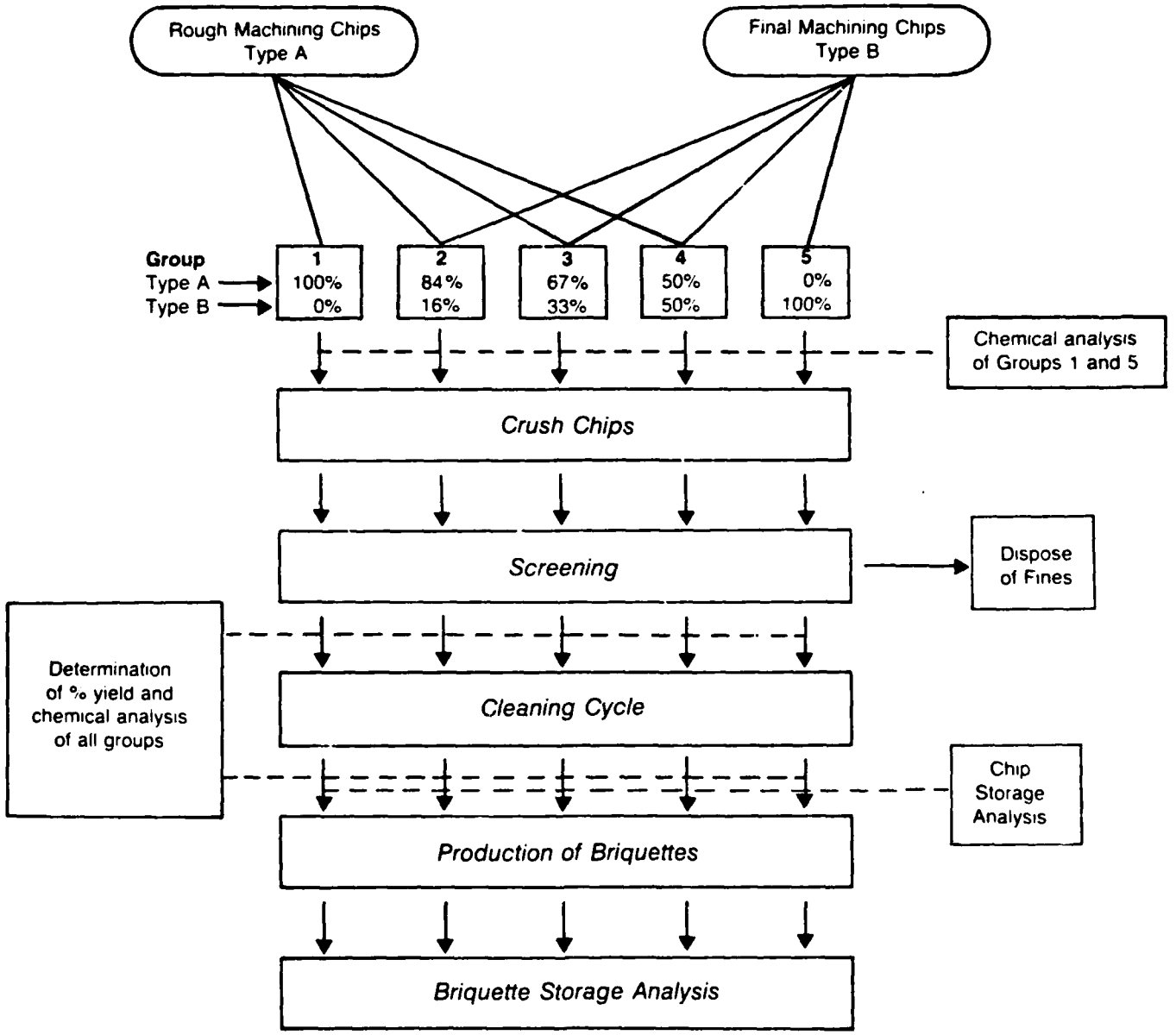


Figure 1 **Flow diagram illustrating formation of test groups and processing steps.**

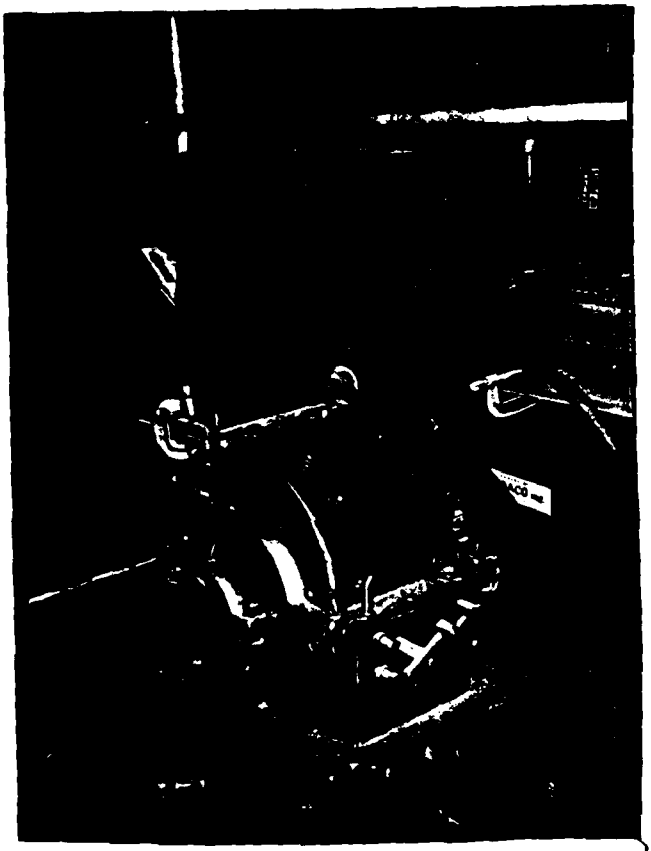


Figure 2. External view of ring crusher.

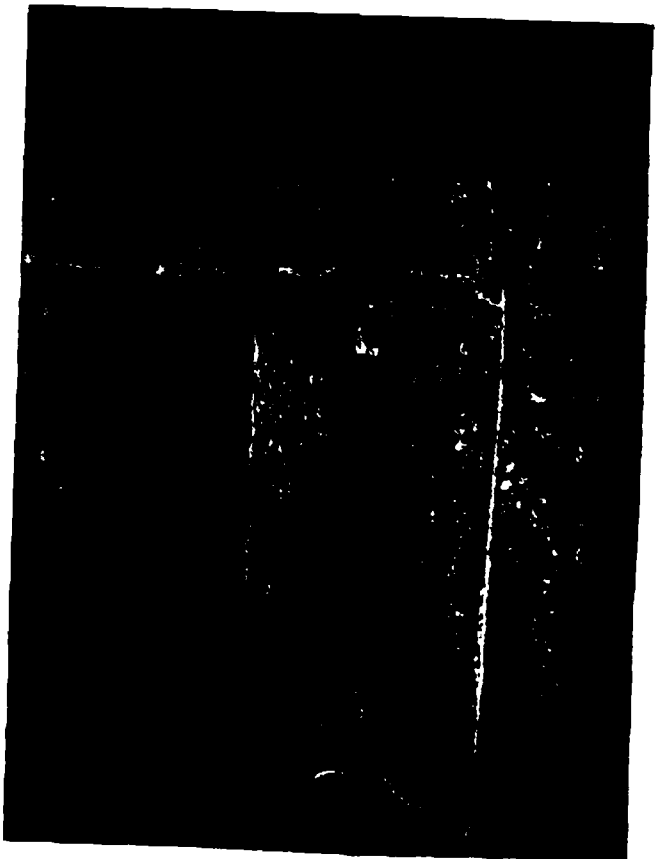


Figure 3. View of actual crushing chips.

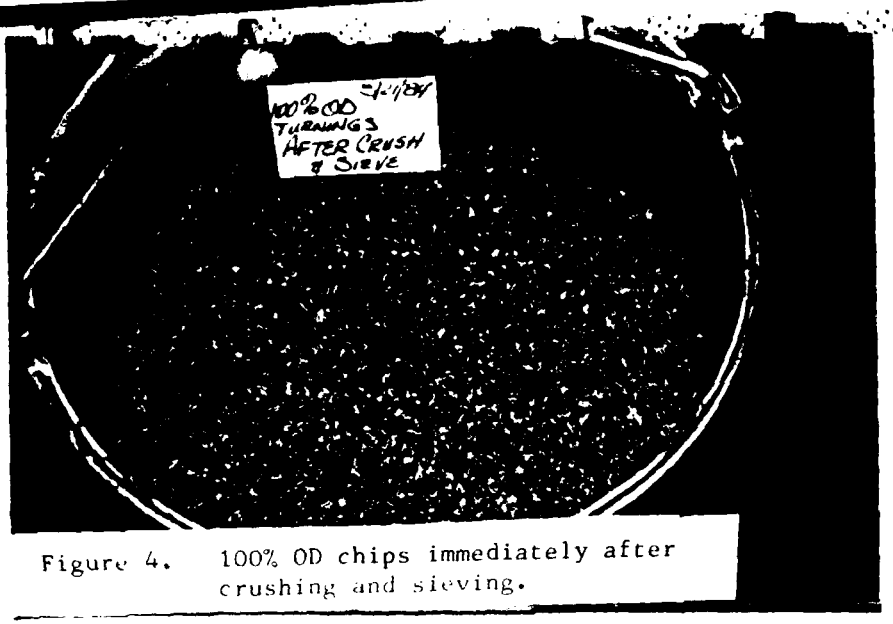


Figure 4. 100% OD chips immediately after crushing and sieving.

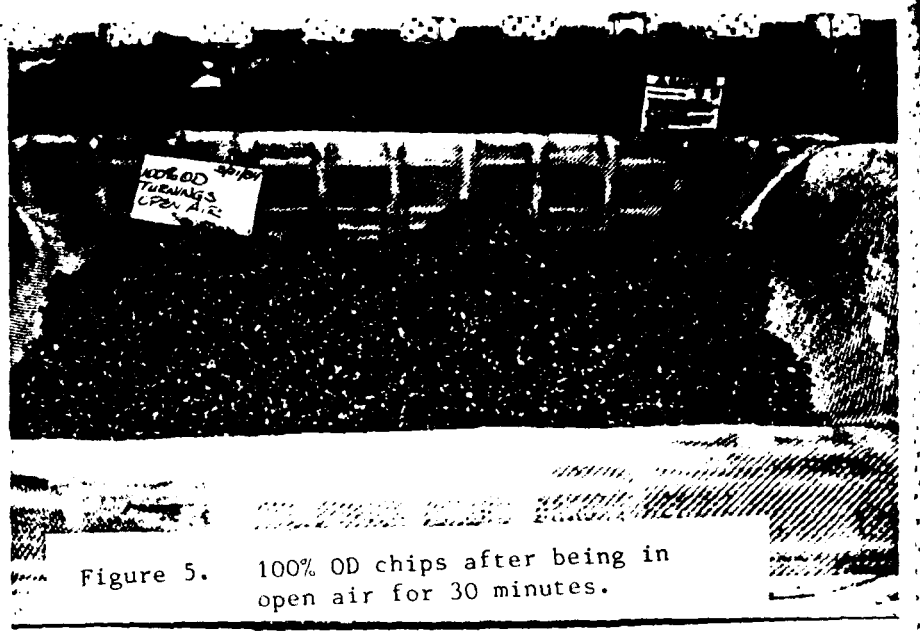


Figure 5. 100% OD chips after being in open air for 30 minutes.

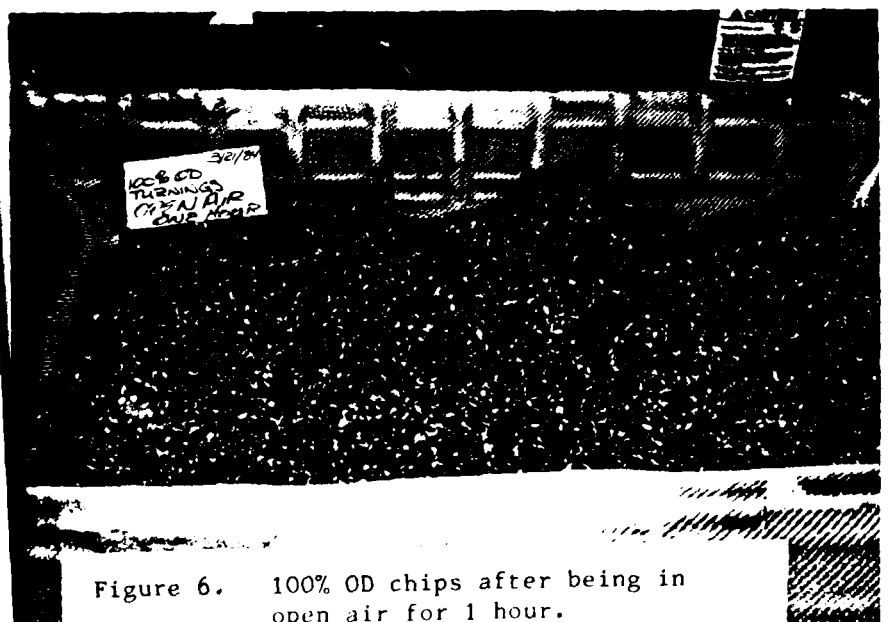


Figure 6. 100% OD chips after being in open air for 1 hour.

Figure 7. 100% OD chips after storage in dry ice and open air for 38 hours.

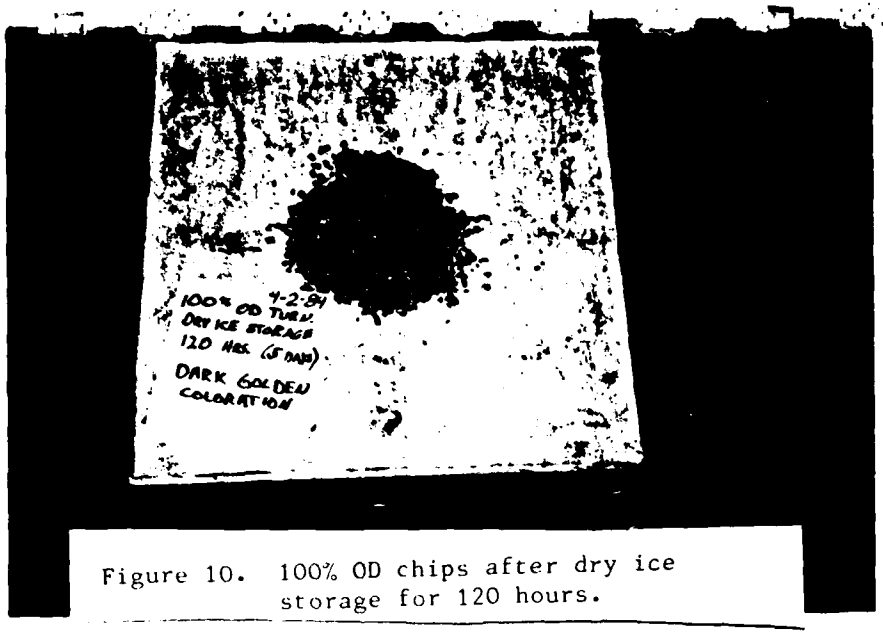


Figure 10. 100% OD chips after dry ice storage for 120 hours.

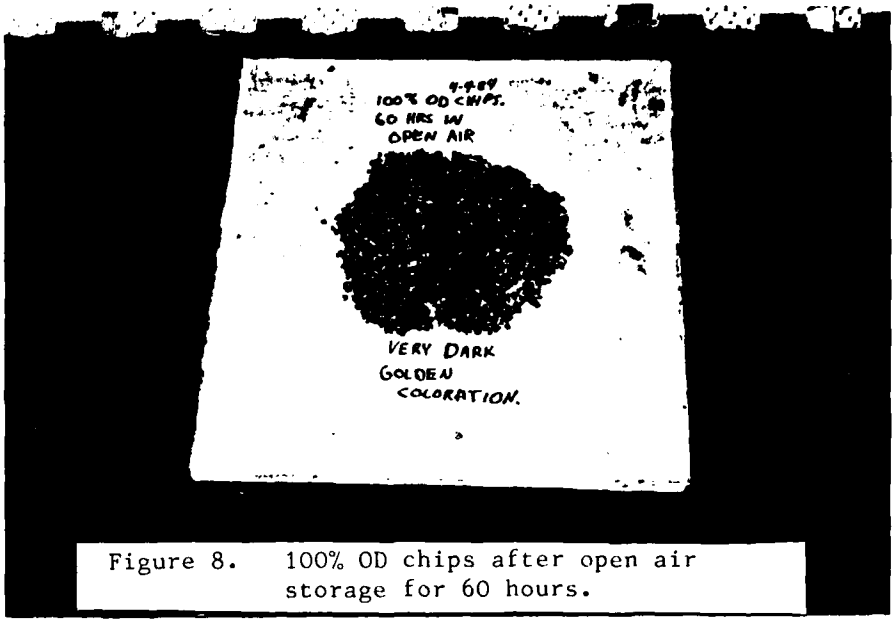


Figure 8. 100% OD chips after open air storage for 60 hours.

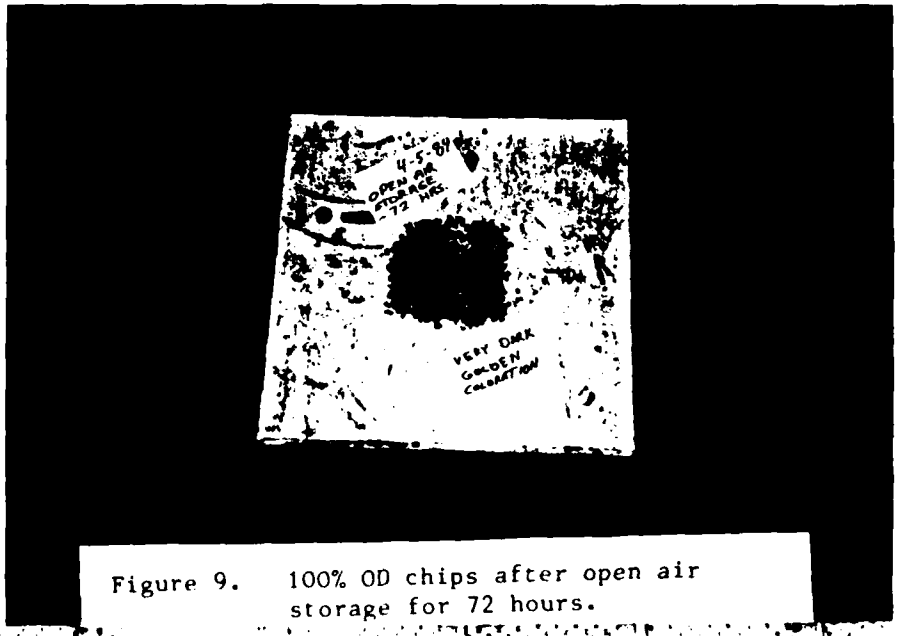


Figure 9. 100% OD chips after open air storage for 72 hours.

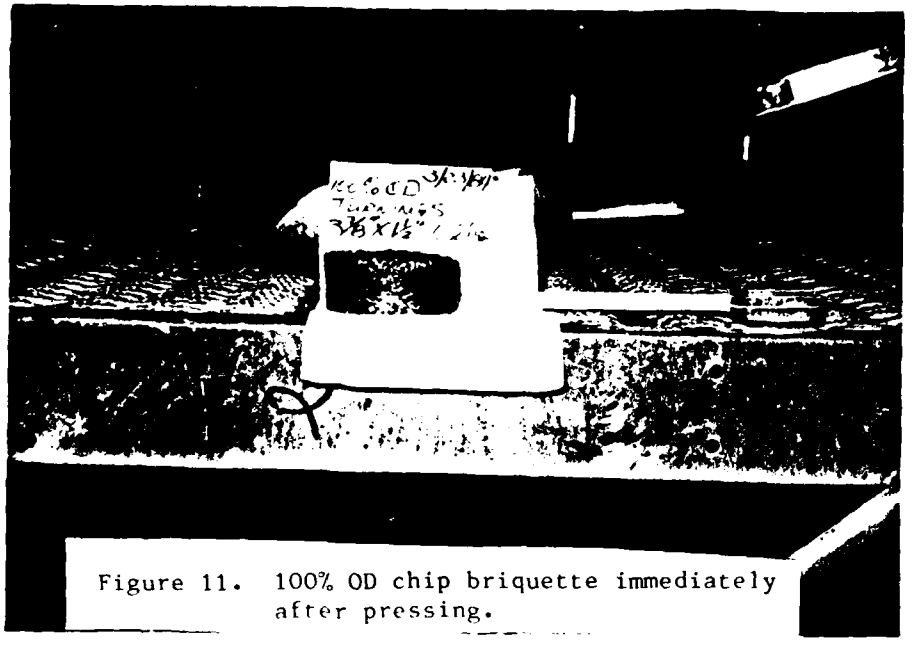


Figure 11. 100% OD chip briquette immediately after pressing.

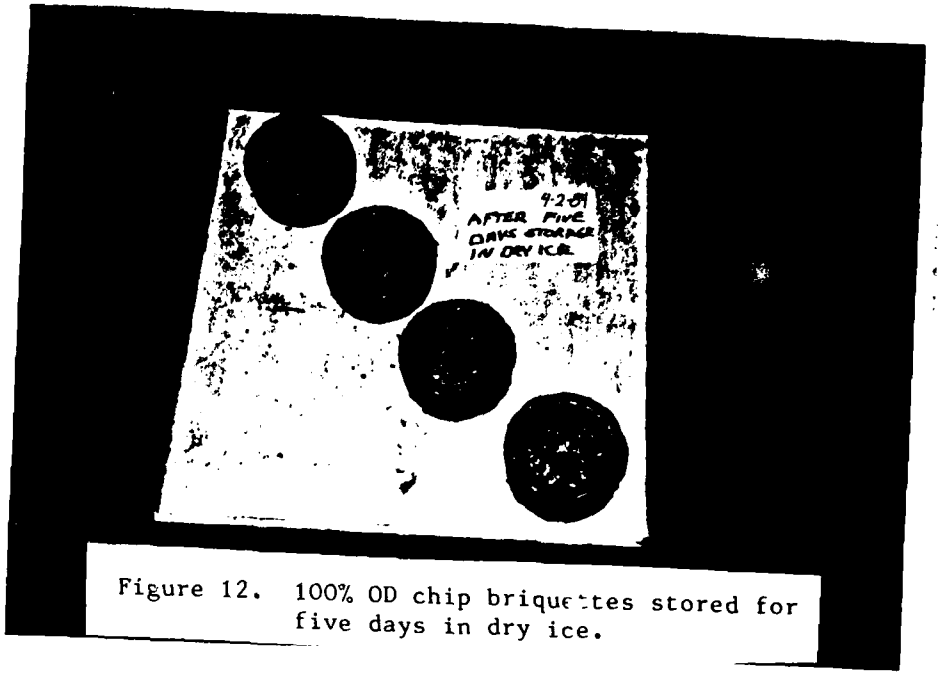


Figure 12. 100% OD chip briquettes stored for five days in dry ice.

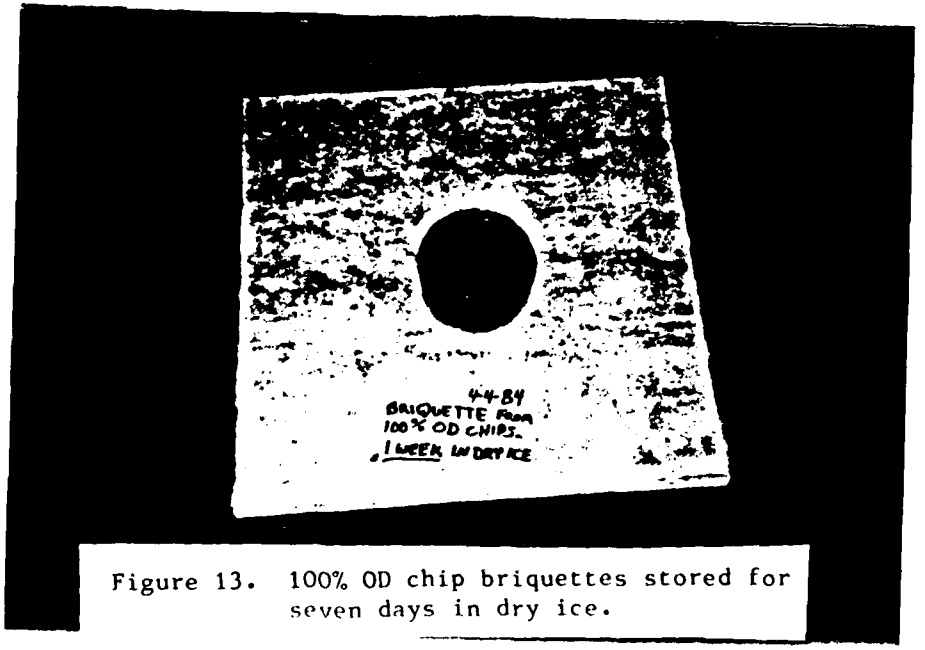


Figure 13. 100% OD chip briquettes stored for seven days in dry ice.

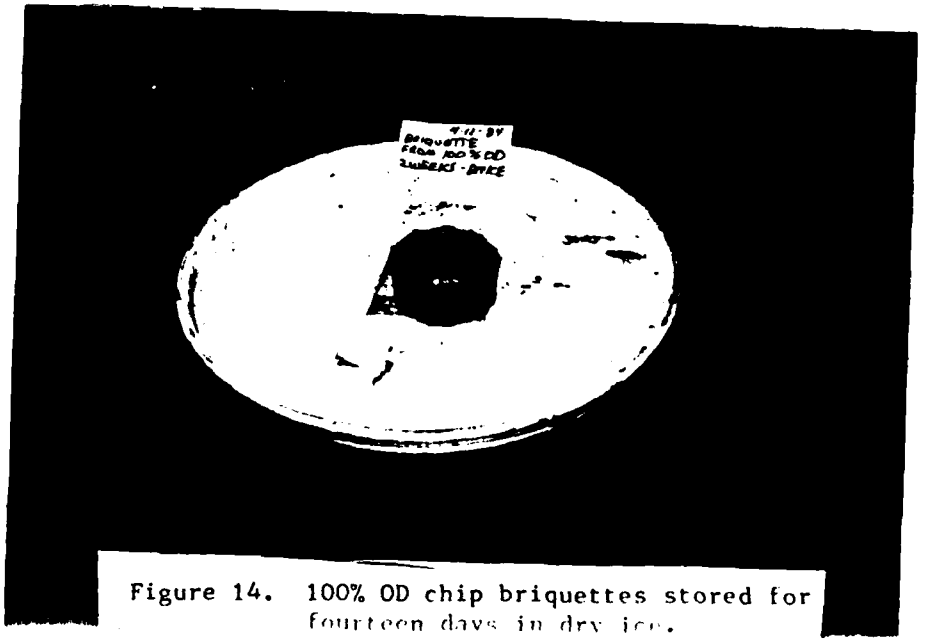


Figure 14. 100% OD chip briquettes stored for fourteen days in dry ice.

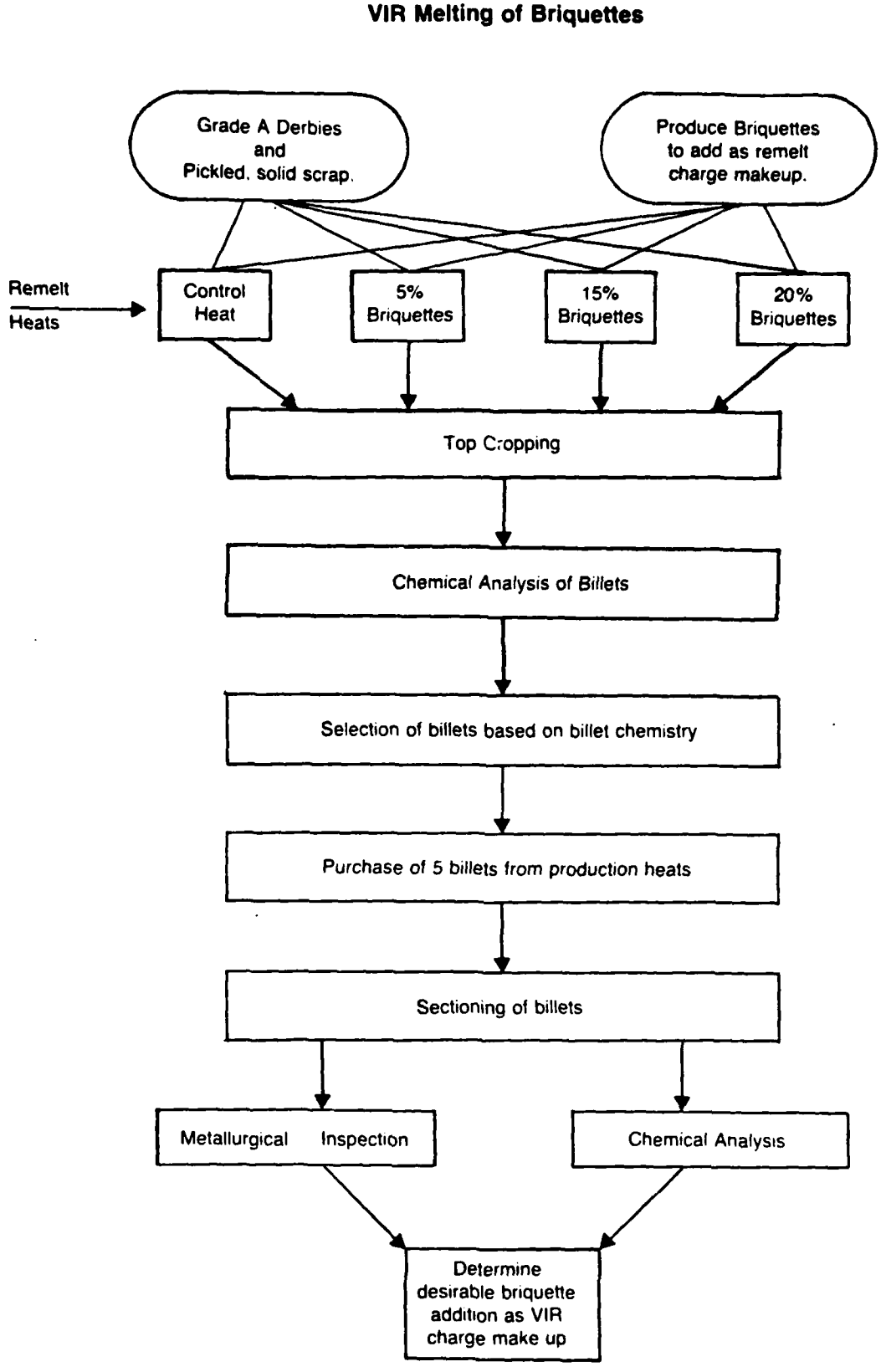


Figure 15 **Flow diagram illustrating VIR charge constituents and subsequent material analysis**

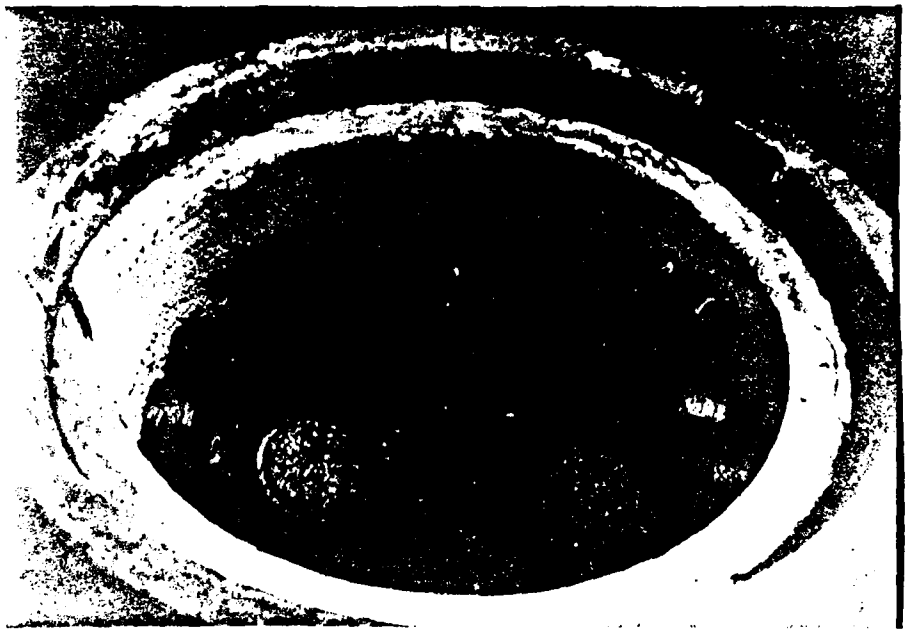


Figure 16. 15" Briquette Charge Crucible Makeup.

Figure 17

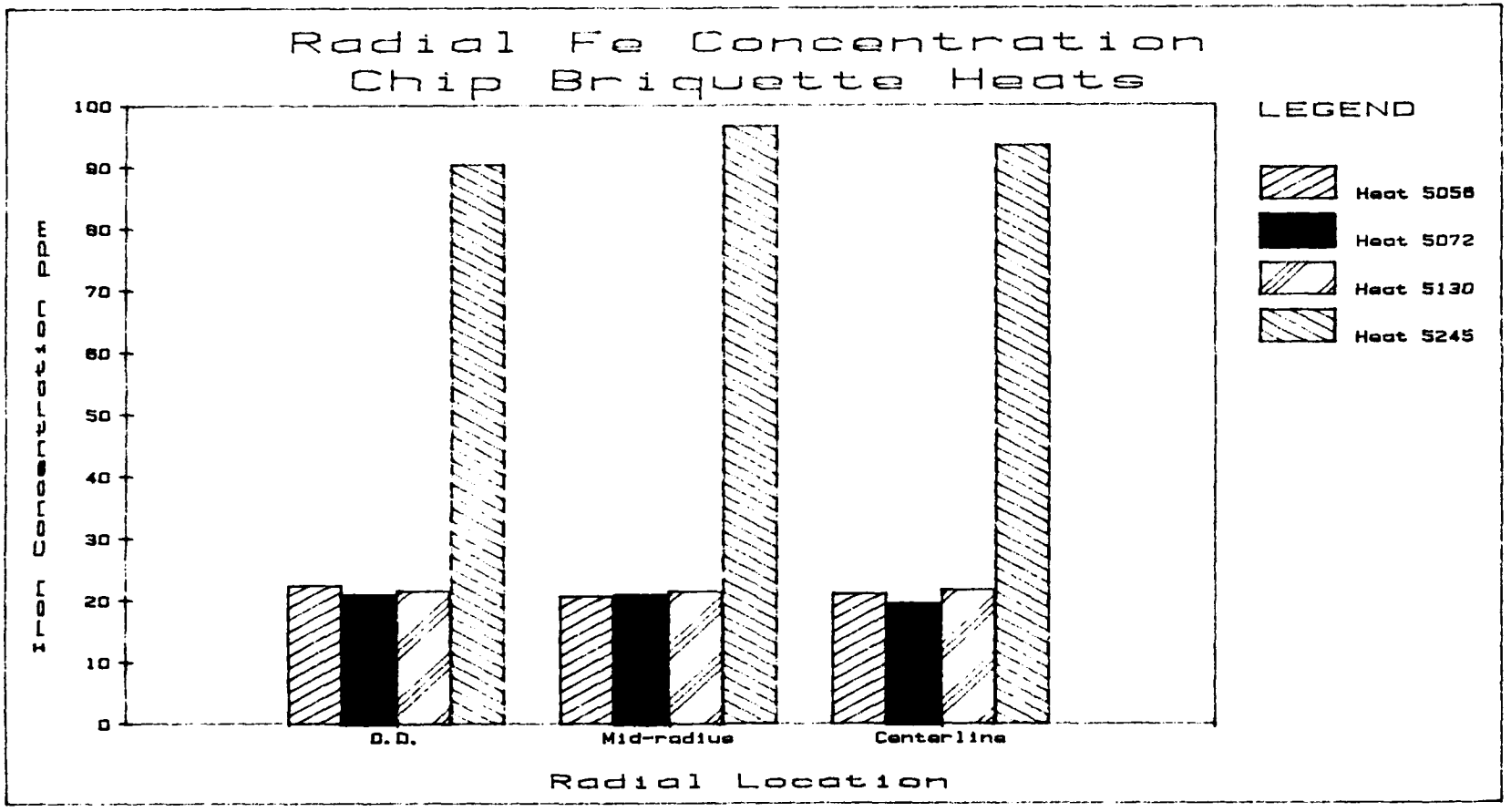
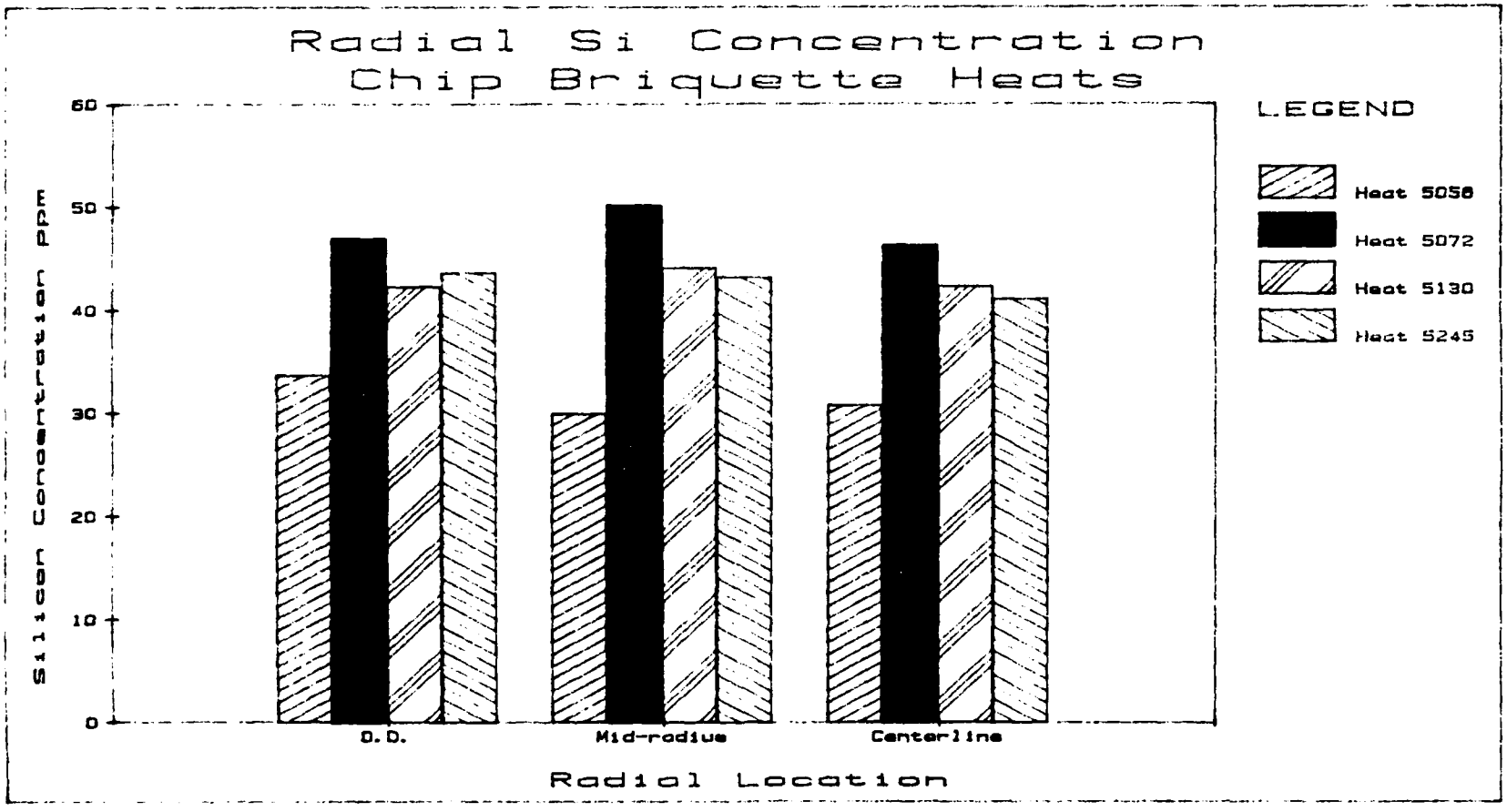

Radial Fe Concentration
Chip Briquette Heats
Iron Concentration ppm
100
90
80
70
60
50
40
30
20
10
0
O.D.
Mid-radius
Centerline
Radial Location
LEGEND
Heat 5058
Heat 5072
Heat 5130
Heat 5245

Figure 18


Radial Si Concentration
Chip Briquette Heats
Silicon Concentration ppm
60
50
40
30
20
10
0
O.D.
Mid-radius
Centerline
Radial Location
LEGEND
Heat 5058
Heat 5072
Heat 5130
Heat 5245

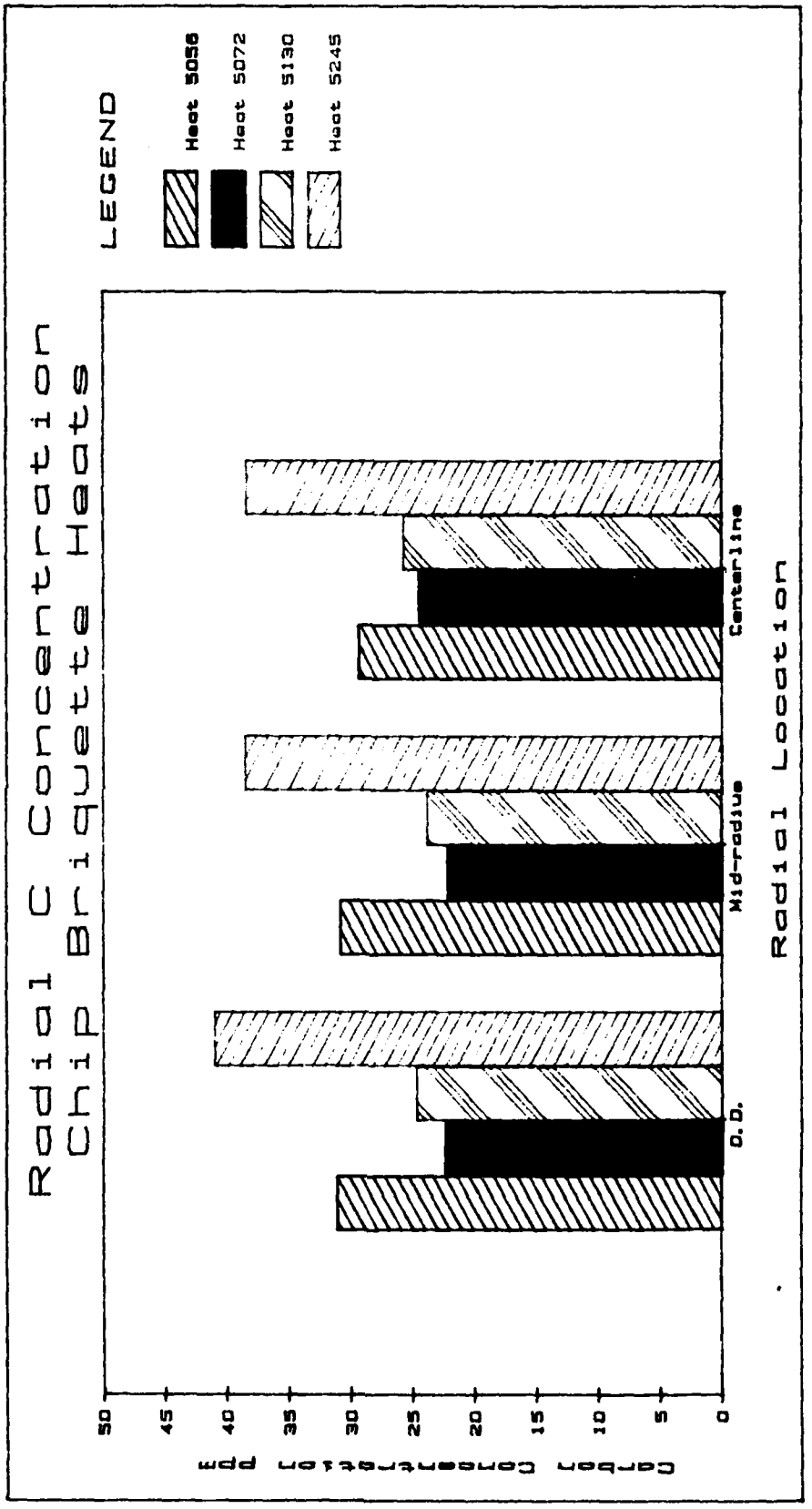

Radial C Concentration
Chip Briquete Heats
LEGEND
Heat 5056
Heat 5072
Heat 5130
Heat 5245
Carbon Concentration ppm
0
5
10
15
20
25
30
35
40
45
50
O.D.
Mid-radius
Centerline
Radial Location

Figure 19

Figure 20

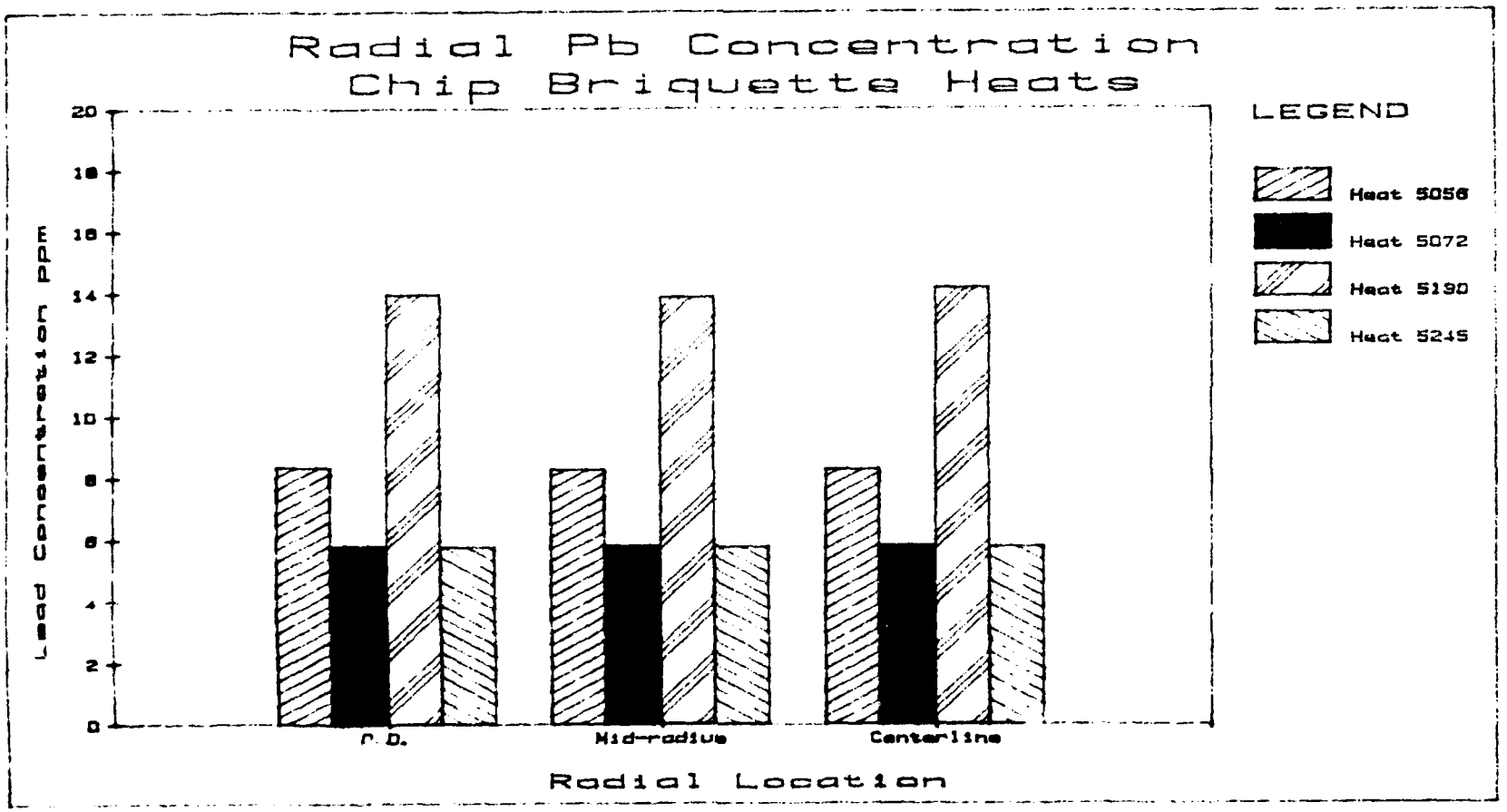
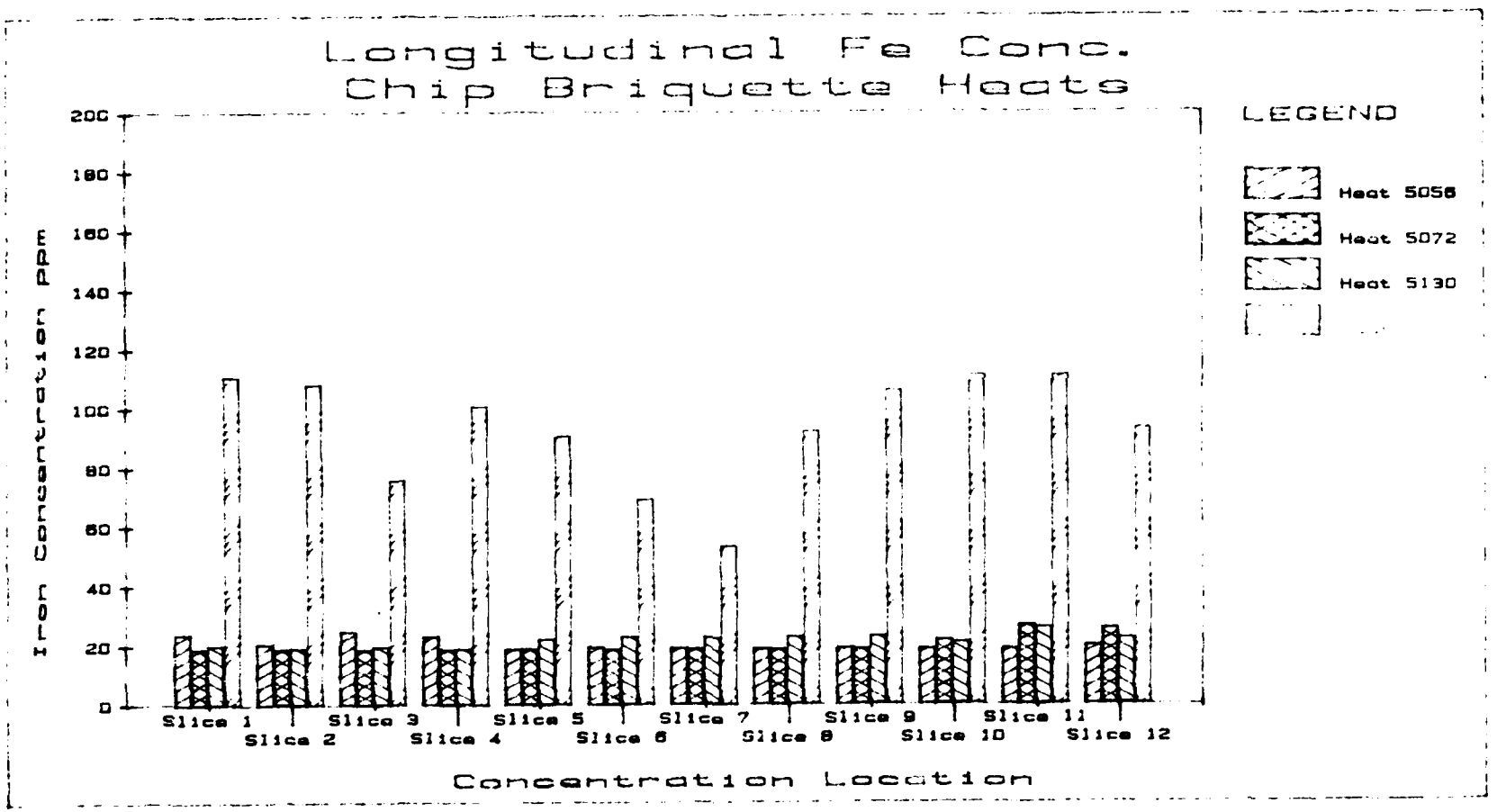

Radial Pb Concentration
Chip Briquette Heats
LEGEND
Heat 5056
Heat 5072
Heat 5190
Heat 5245
Lead Concentration ppm
0
2
4
6
8
10
12
14
16
18
20
O.D.
Mid-radius
Centerline
Radial Location

Figure 21


Longitudinal Fe Conc.
Chip Briquette Heats
Iron Concentration ppm
200
180
160
140
120
100
80
60
40
20
0
Slice 1
Slice 2
Slice 3
Slice 4
Slice 5
Slice 6
Slice 7
Slice 8
Slice 9
Slice 10
Slice 11
Slice 12
Concentration Location
LEGEND
Heat 5056
Heat 5072
Heat 5130

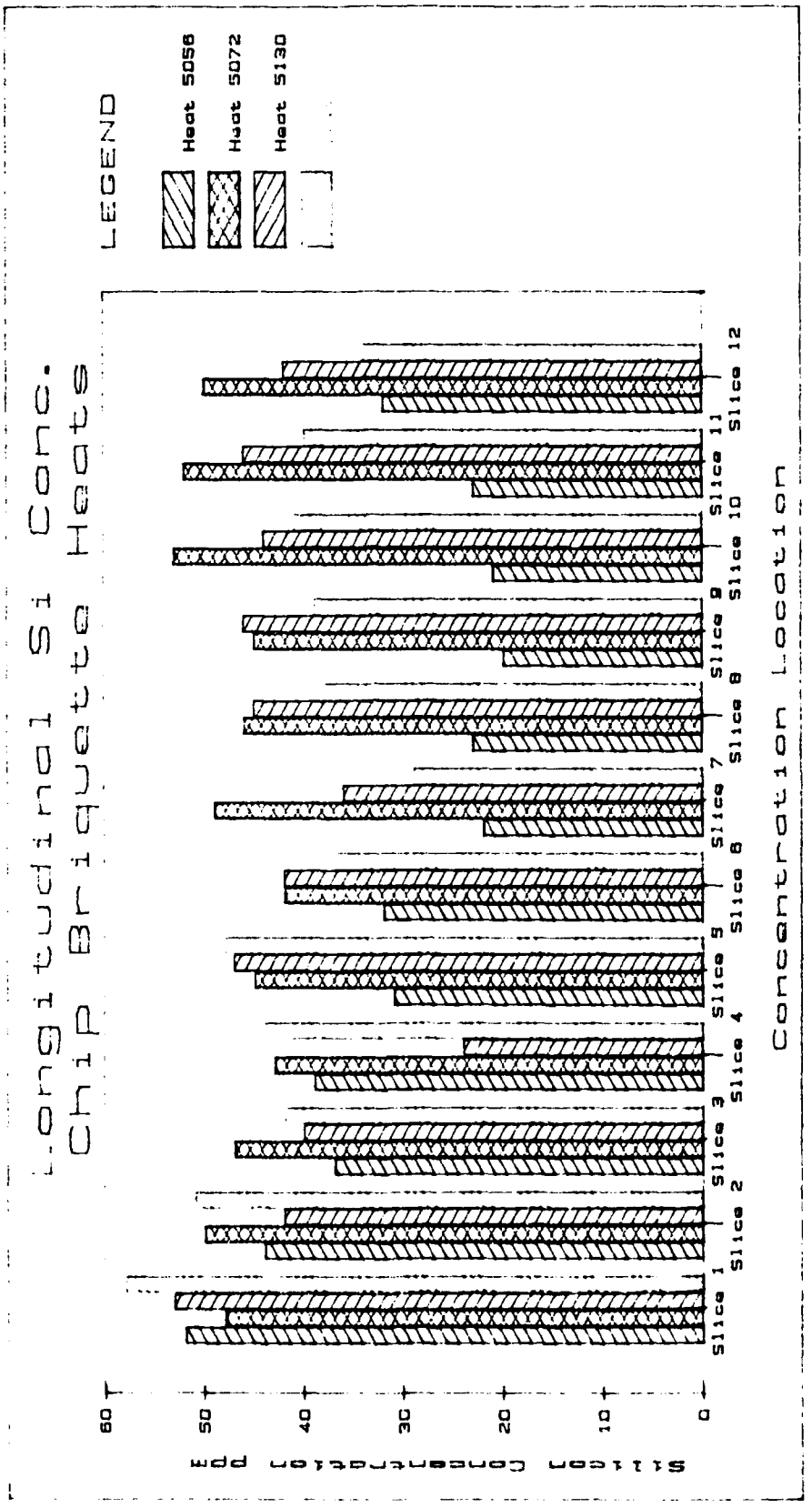
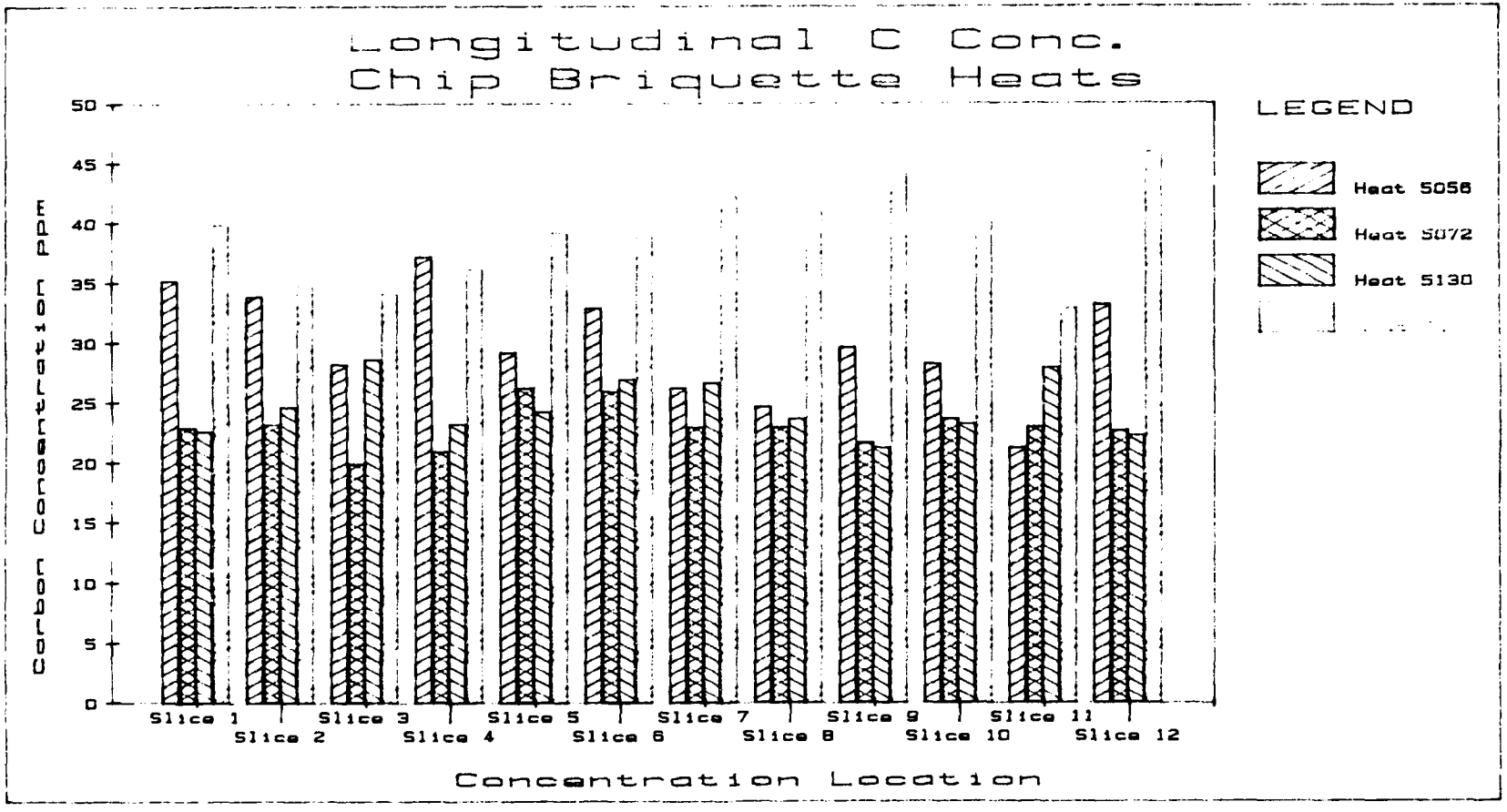

Longitudinal Si Conc.
Chip Briquette Heats
LEGEND
Heat 5056
Heat 5072
Heat 5130
Silicon Concentration ppm
60
50
40
30
20
10
0
Slice 1
Slice 2
Slice 3
Slice 4
Slice 5
Slice 6
Slice 7
Slice 8
Slice 9
Slice 10
Slice 11
Slice 12
Concentration Location

Figure 22

Figure 23


Longitudinal C Conc.
Chip Briquette Heats
LEGEND
Heat 5056
Heat 5072
Heat 5130
Carbon Concentration ppm
0
5
10
15
20
25
30
35
40
45
50
Slice 1
Slice 2
Slice 3
Slice 4
Slice 5
Slice 6
Slice 7
Slice 8
Slice 9
Slice 10
Slice 11
Slice 12
Concentration Location

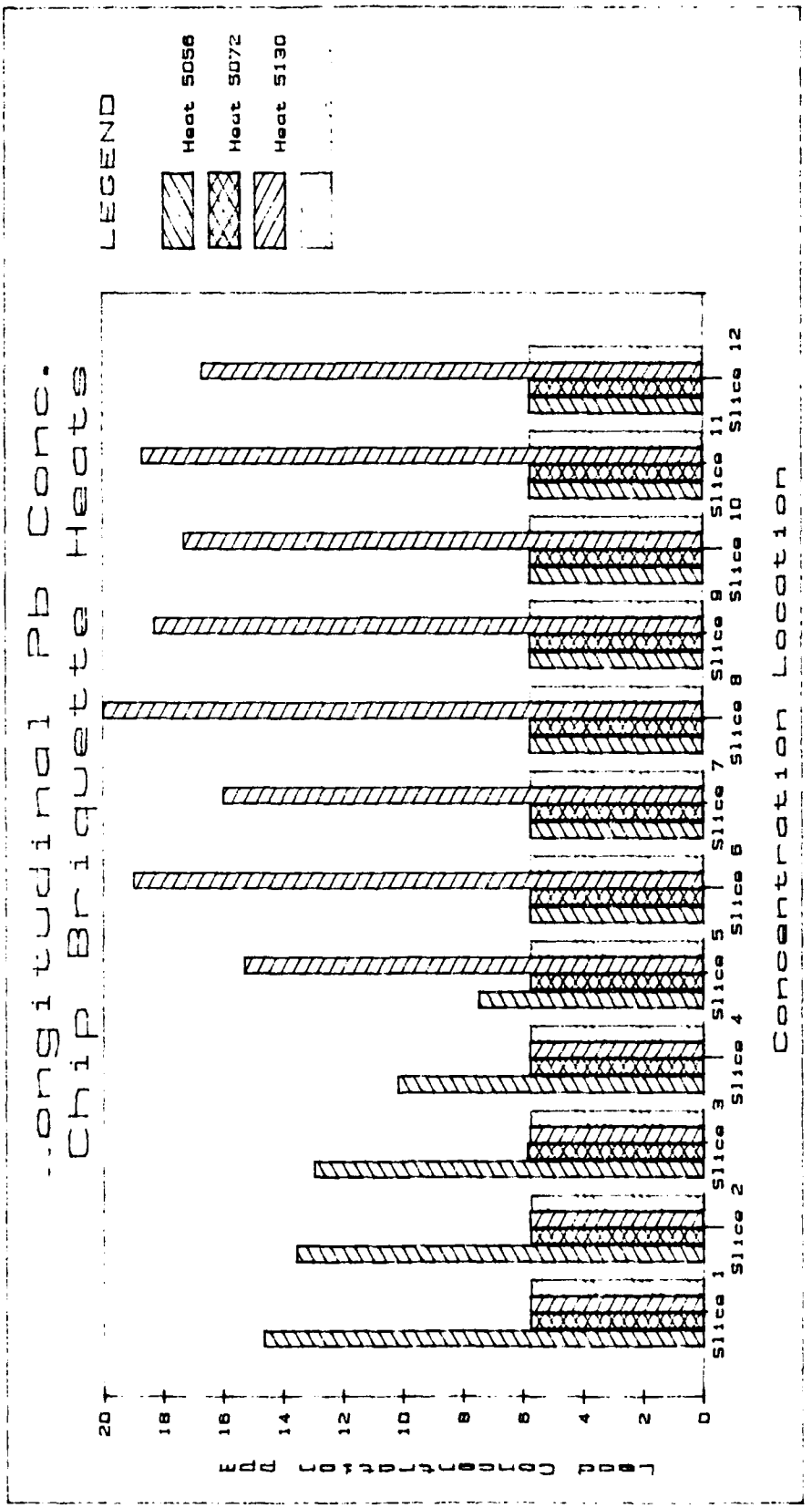
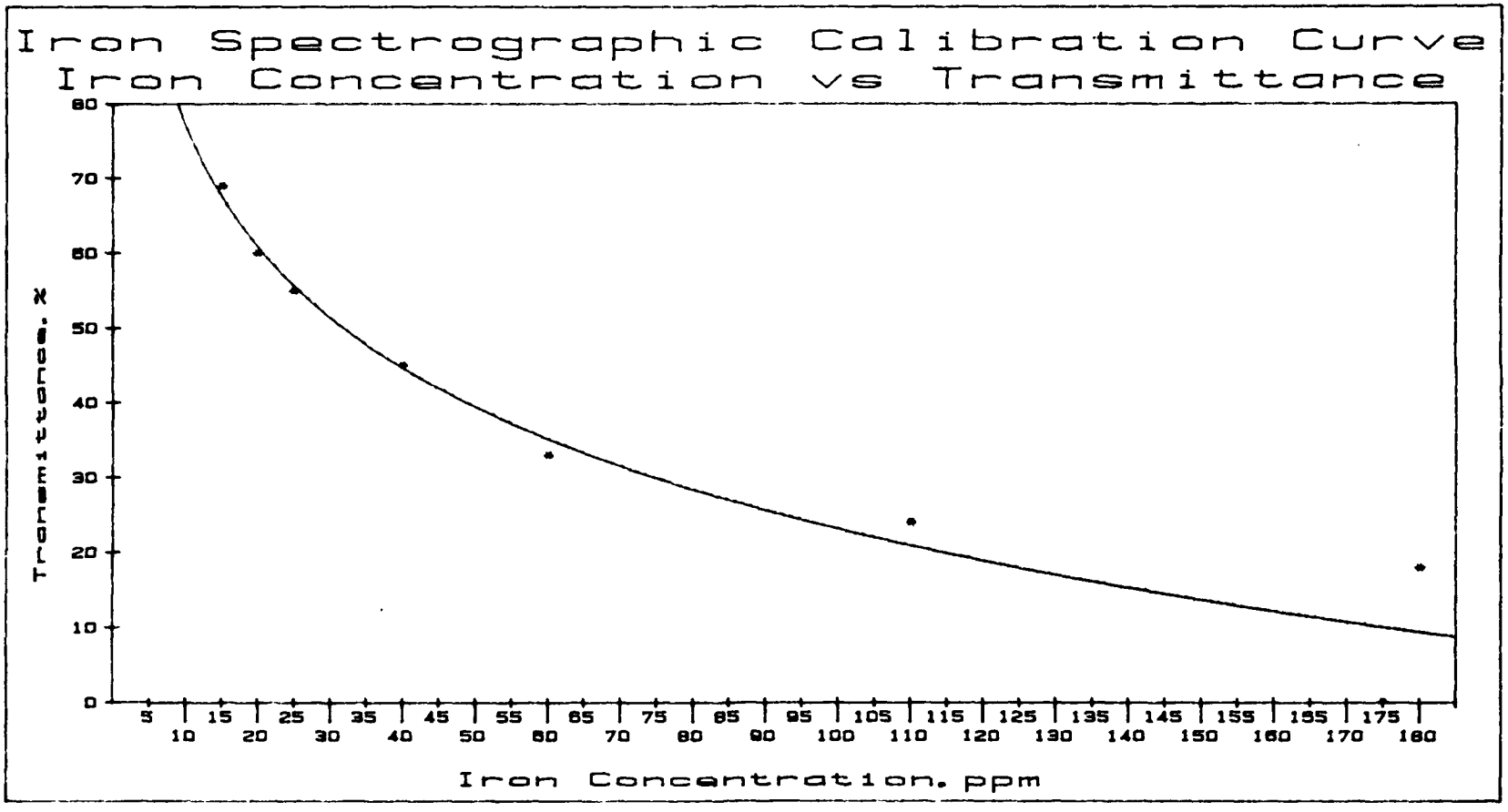

Longitudinal Pb Conc.
Chip Briquette Heats
LEGEND
Heat 5056
Heat 9072
Heat 5130
Lead Concentration ppm
0
2
4
6
8
10
12
14
16
18
20
Slice 1
Slice 2
Slice 3
Slice 4
Slice 5
Slice 6
Slice 7
Slice 8
Slice 9
Slice 10
Slice 11
Slice 12
Concentration Location

Figure 24

Figure 25


Iron Spectrographic Calibration Curve
Iron Concentration vs Transmittance
Transmittance, %
0
10
20
30
40
50
60
70
80
5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105 110 115 120 125 130 135 140 145 150 155 160 165 170 175 180
Iron Concentration, ppm

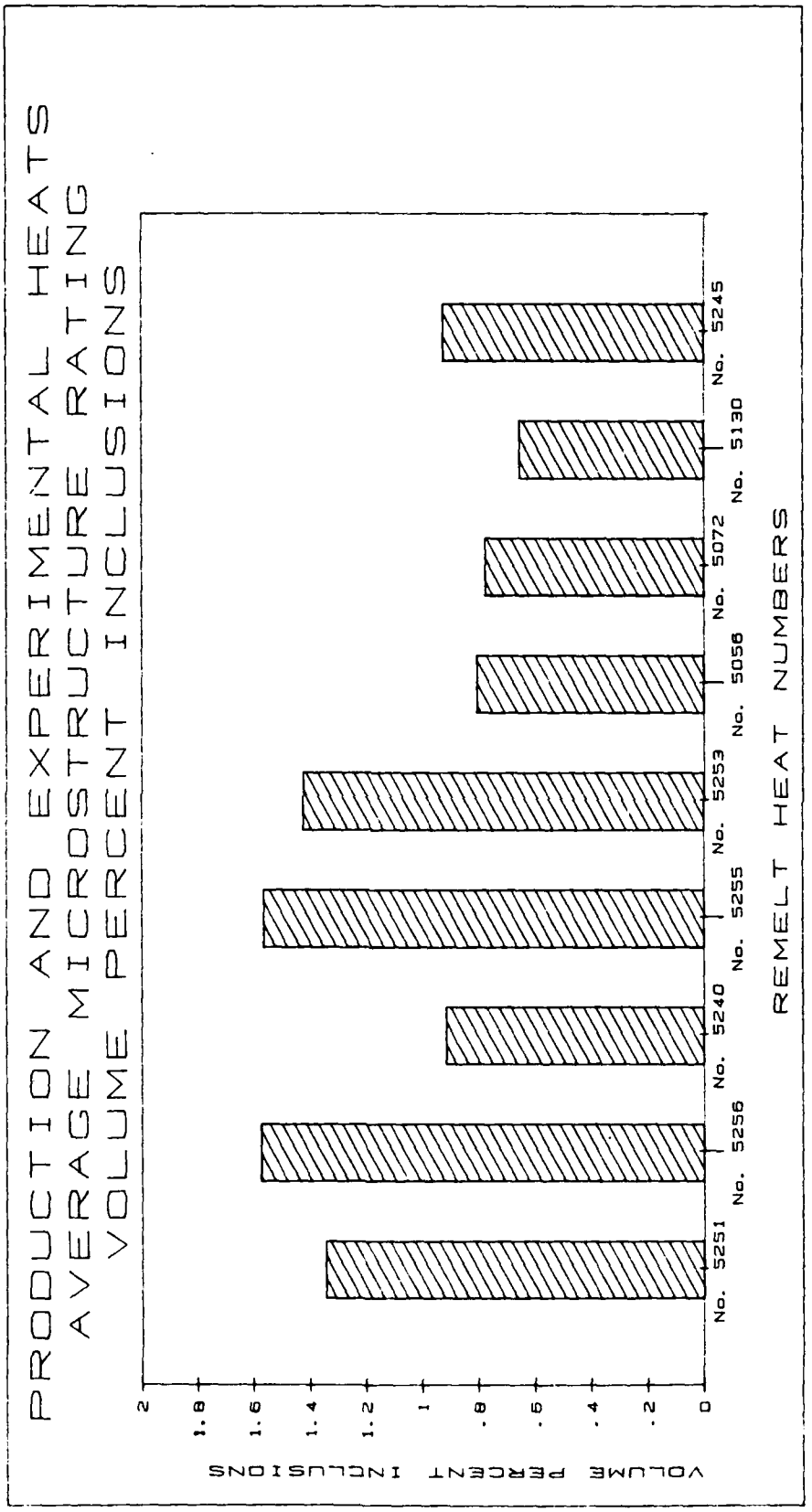

PRODUCTION AND EXPERIMENTAL HEATS
AVERAGE MICROSTRUCTURE RATING
VOLUME PERCENT INCLUSIONS
VOLUME PERCENT INCLUSIONS
0
.2
.4
.6
.8
1
1.2
1.4
1.6
1.8
2
No. 5251
No. 5256
No. 5240
No. 5255
No. 5253
No. 5056
No. 5072
No. 5130
No. 5245
REMELT HEAT NUMBERS

Figure 26

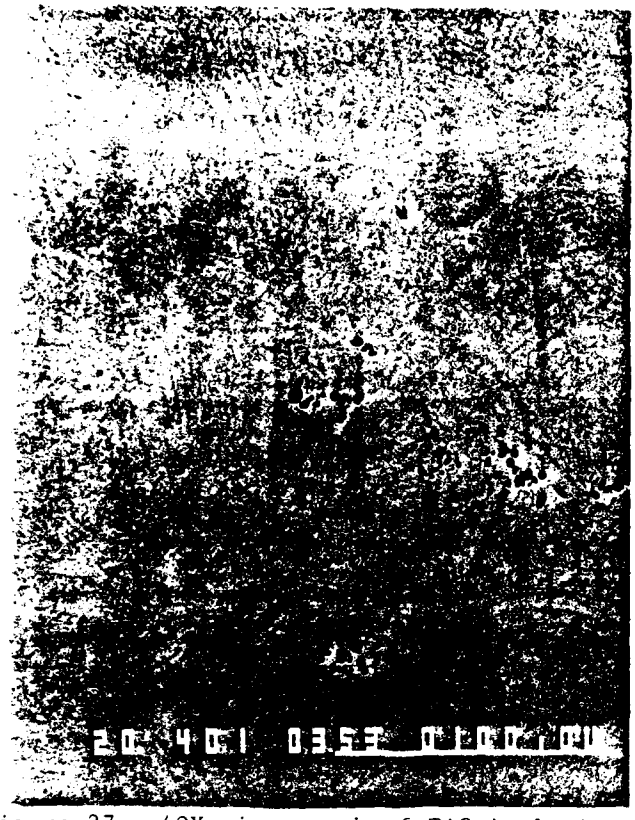


Figure 27. 40X micrograph of TiC inclusion in heat 5245, 20% briquette heat.

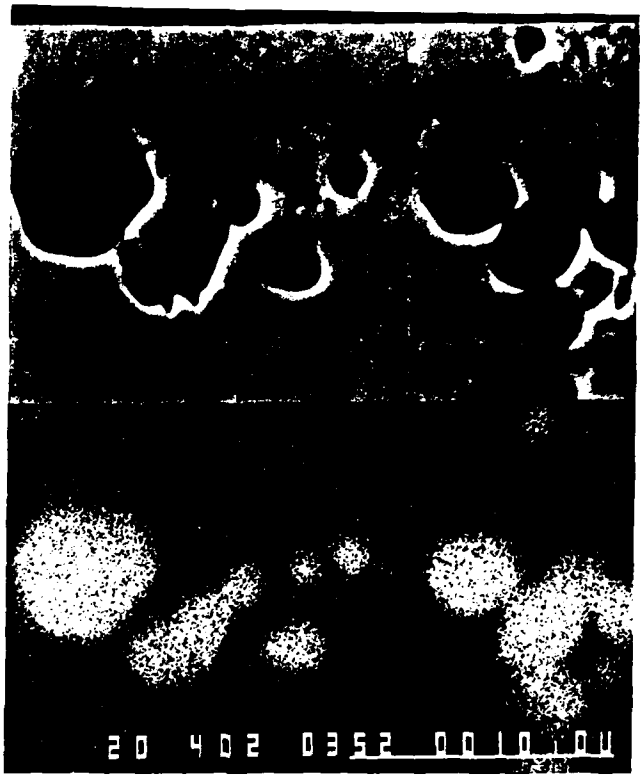


Figure 28. X-ray diffraction pattern for titanium at 400X magnification, heat 5245.

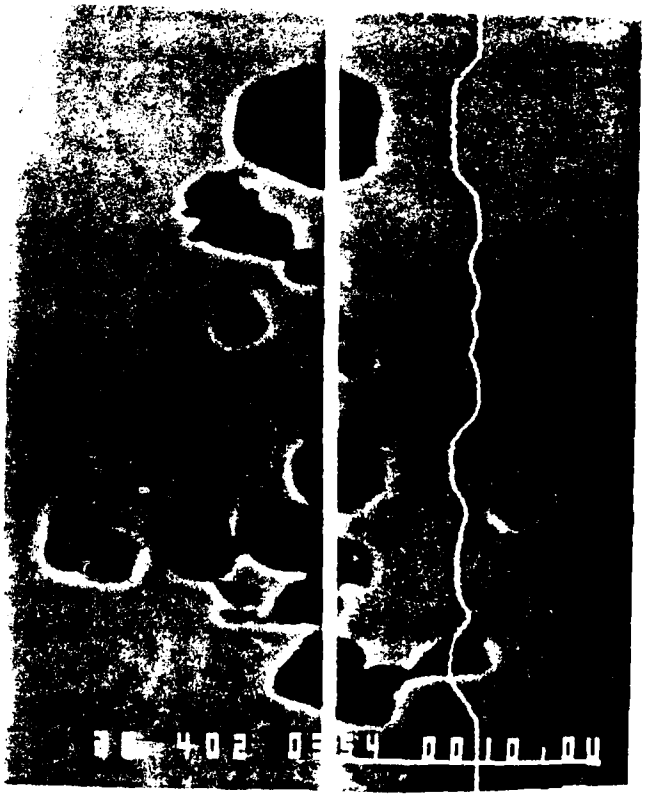


Figure 29. Wavelength dispersive line trace for carbon content, heat 5245.

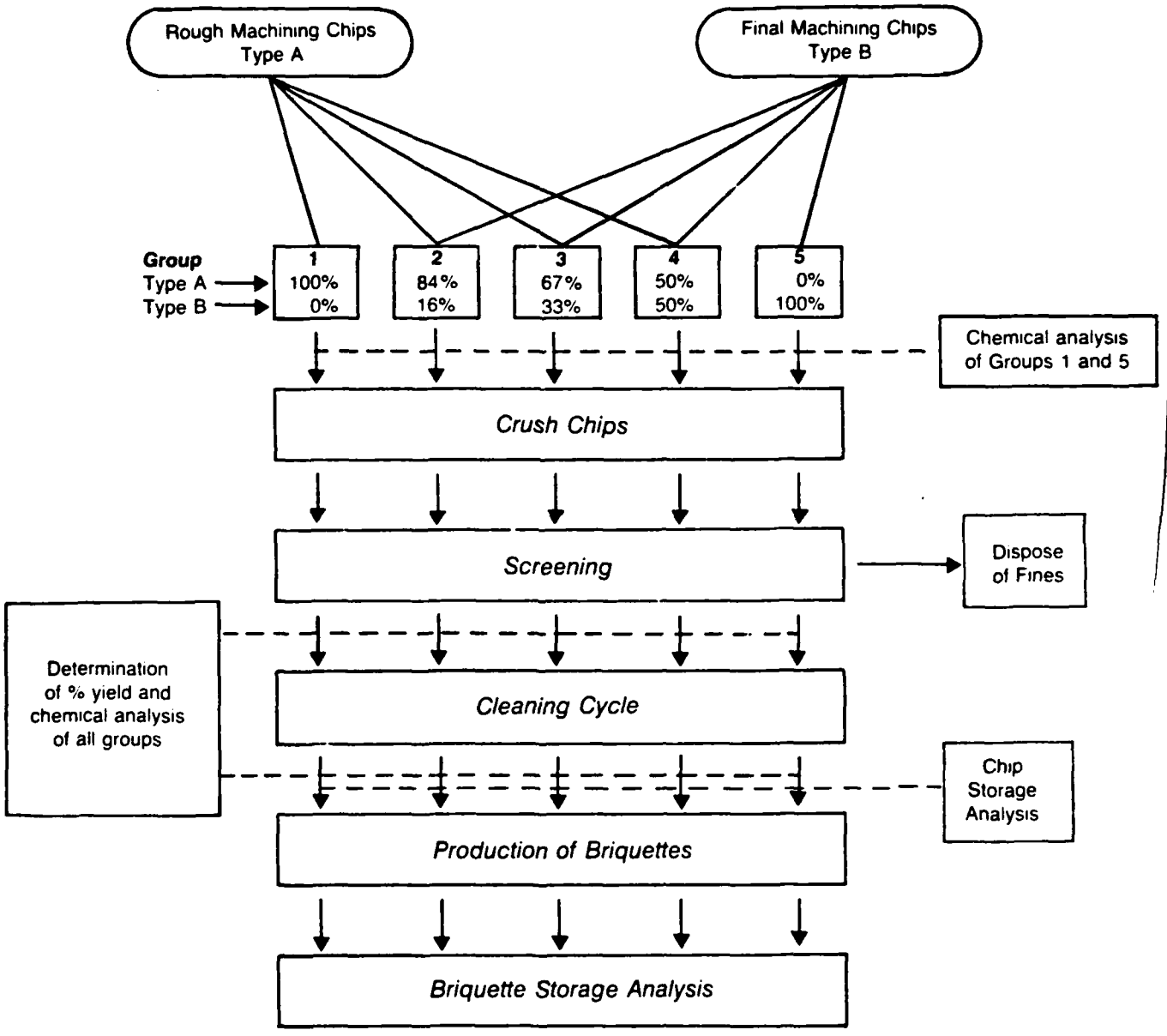


Figure 30 **Flow diagram illustrating formation of test groups and processing steps.**

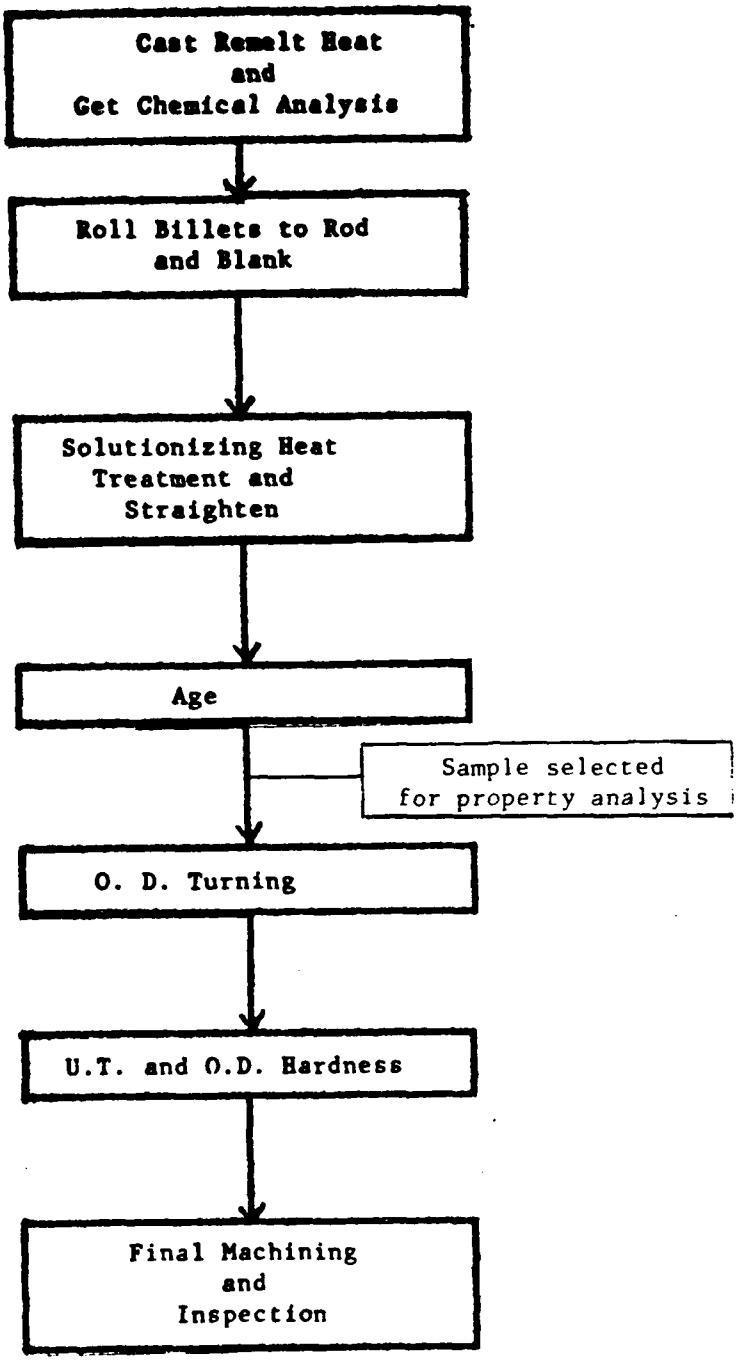
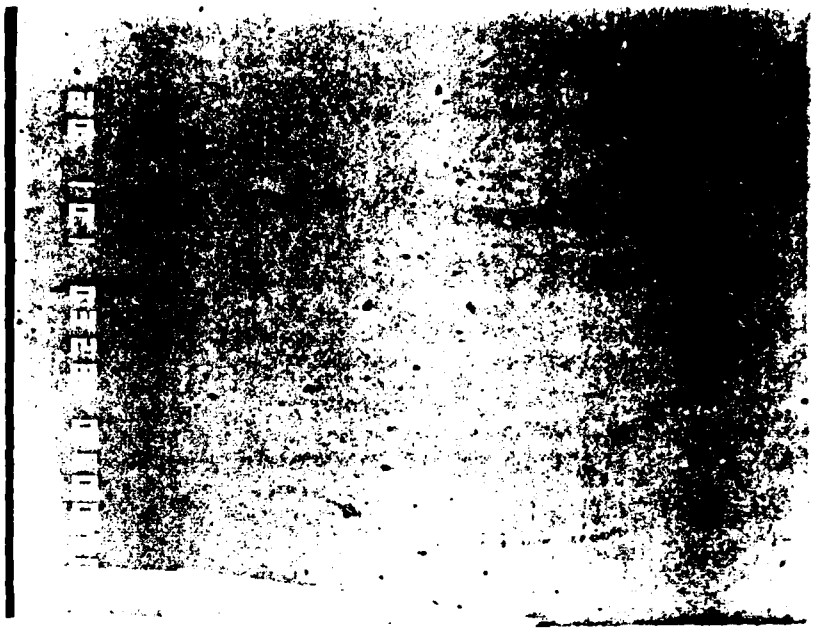


Figure 31 Process 7% Chip Briquette Heat Through to Final Cores

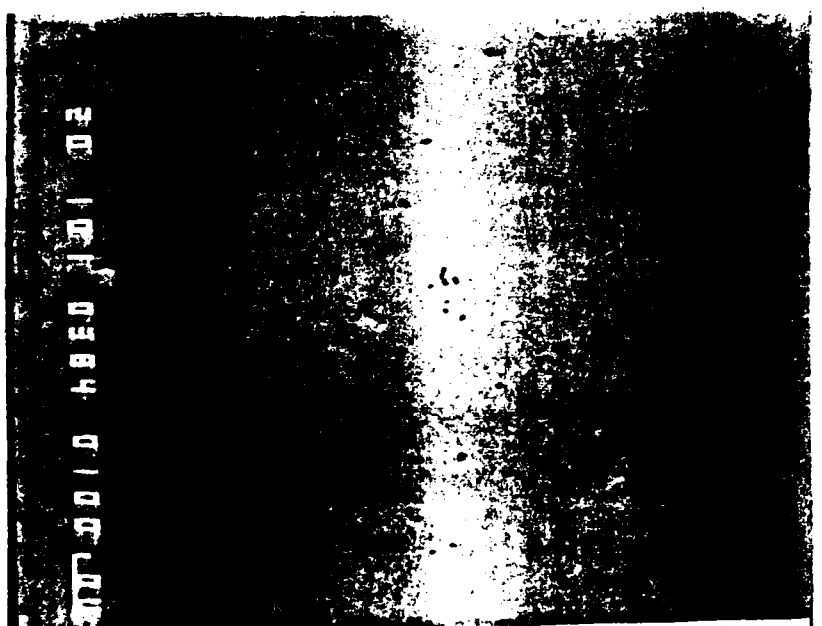
APPENDIX A

MICROGRAPHS OF PRODUCTION AND EXPERIMENTAL BILLET SLICES FOR TASK B

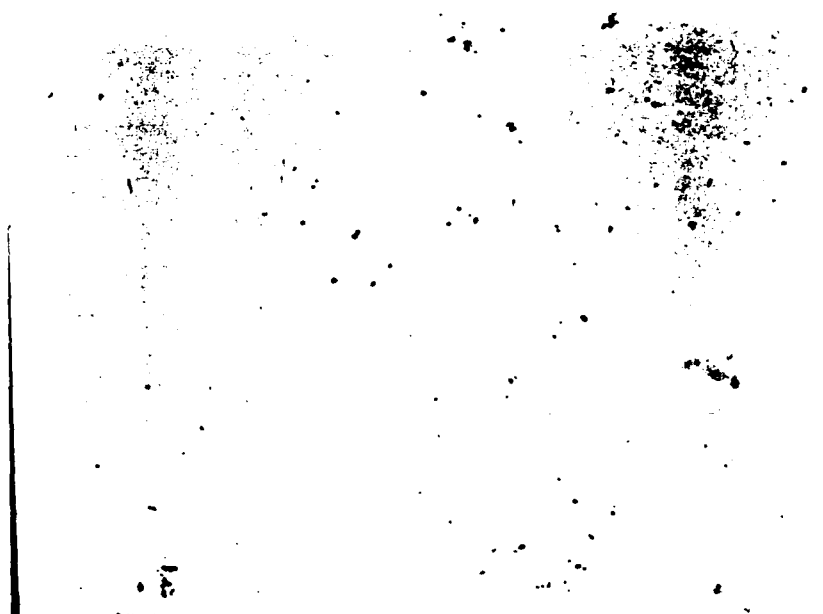
BLANK PAGE



Heat 5240 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X

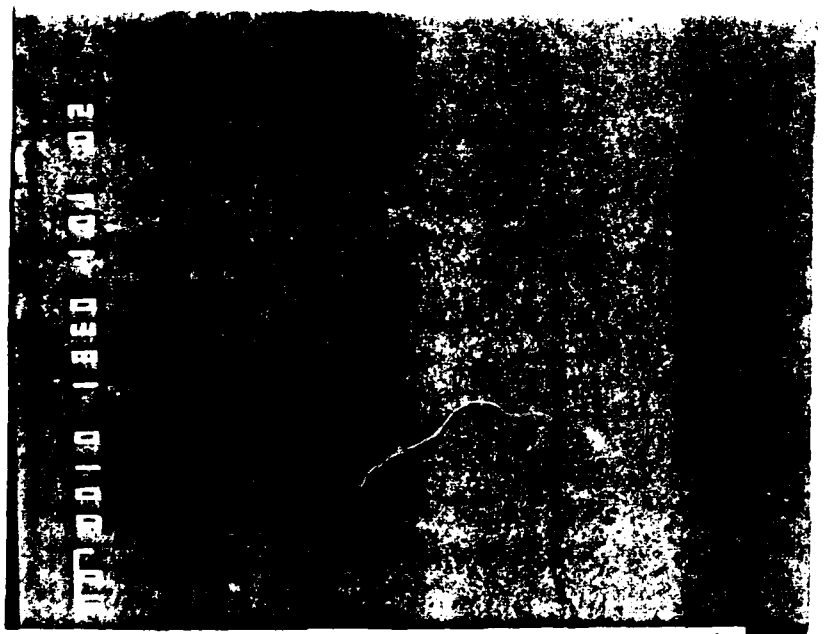


Heat 5240 - Adjacent to the top crop and taken from the centerline location - Transverse 100X

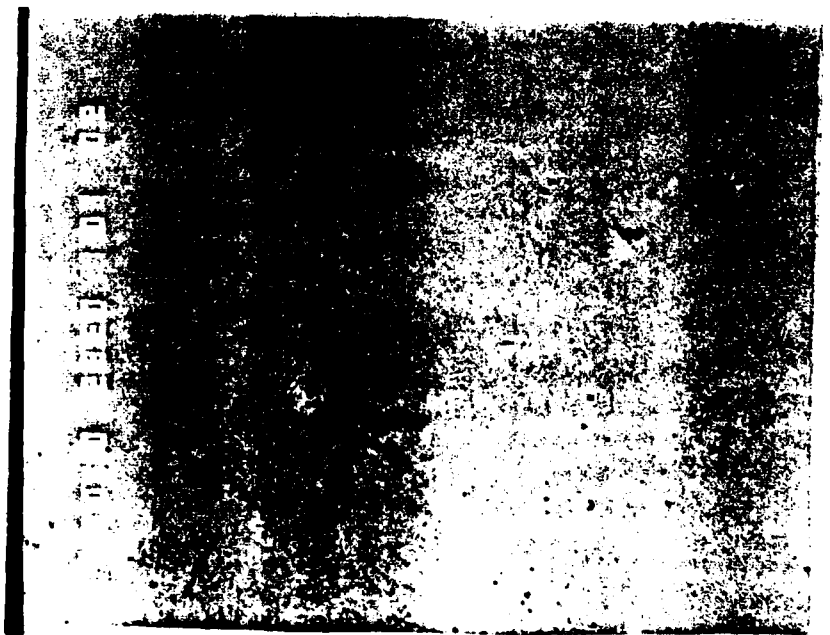


Heat 5240 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X

Heat 5240 - Adjacent to the toe and taken from the O.D. location - Transverse 100X



Heat 5240 - Adjacent to the toe and taken from the centerline location - Transverse 100X



Heat 5240 - Adjacent to the toe and taken from the mid-radius location - Transverse 100X

Heat 5256 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X

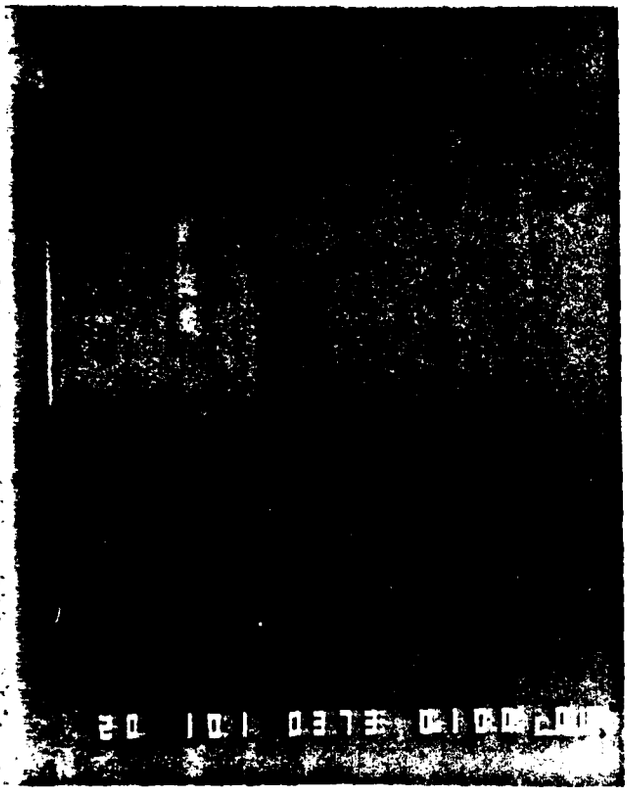
5256LH O

Heat 5256 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X

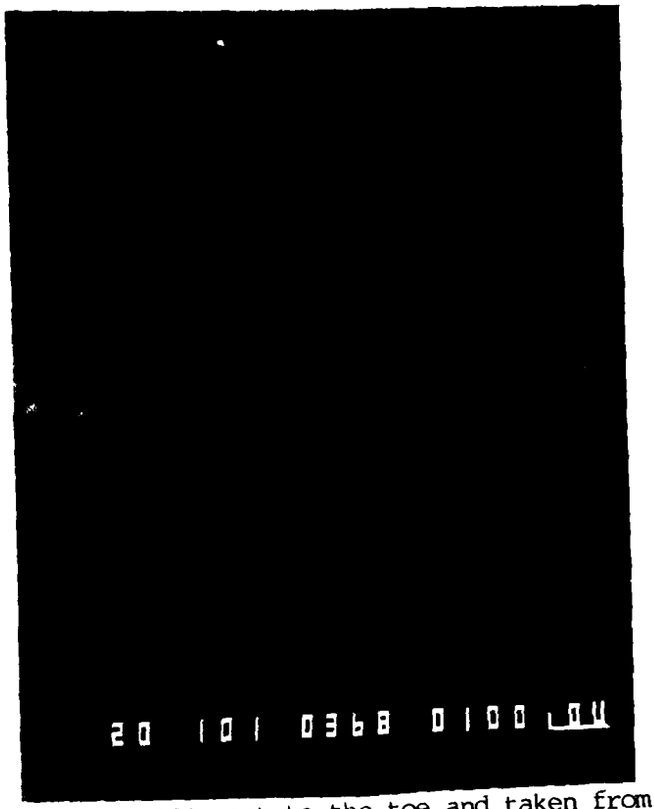
5256LH M

Heat 5256 - Adjacent to the top crop and taken from the centerline location - Transverse 100X

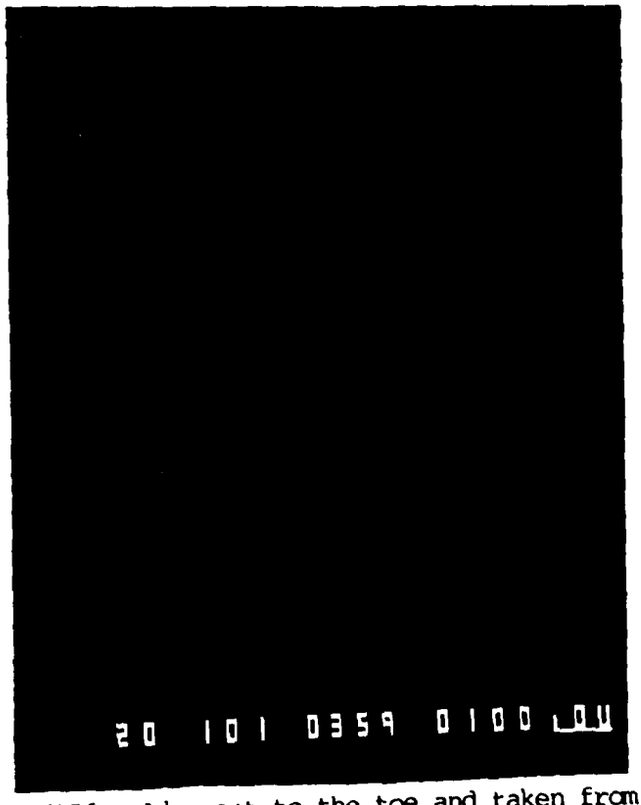
5256LH C



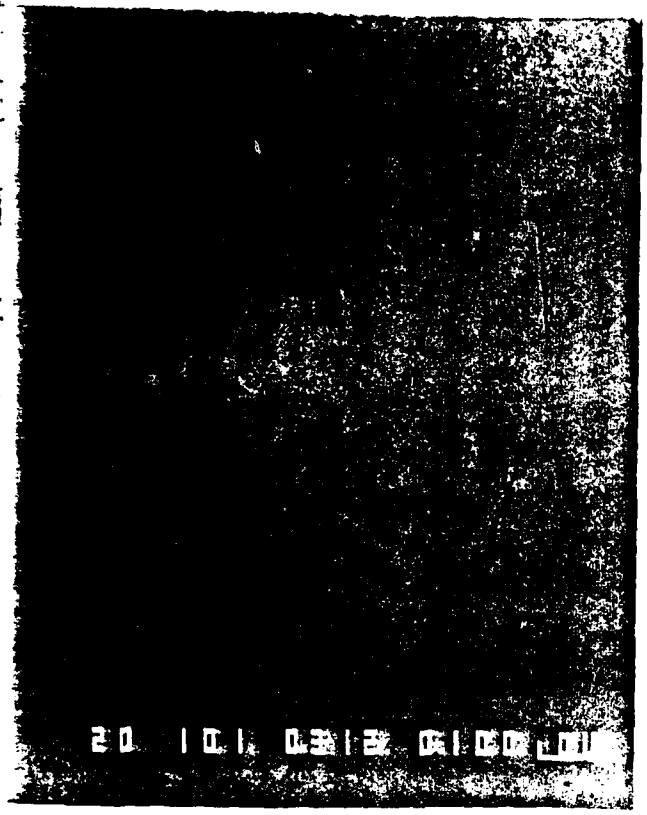
t 5256 - Adjacent to the toe and taken from the O.D. location - Transverse 100X



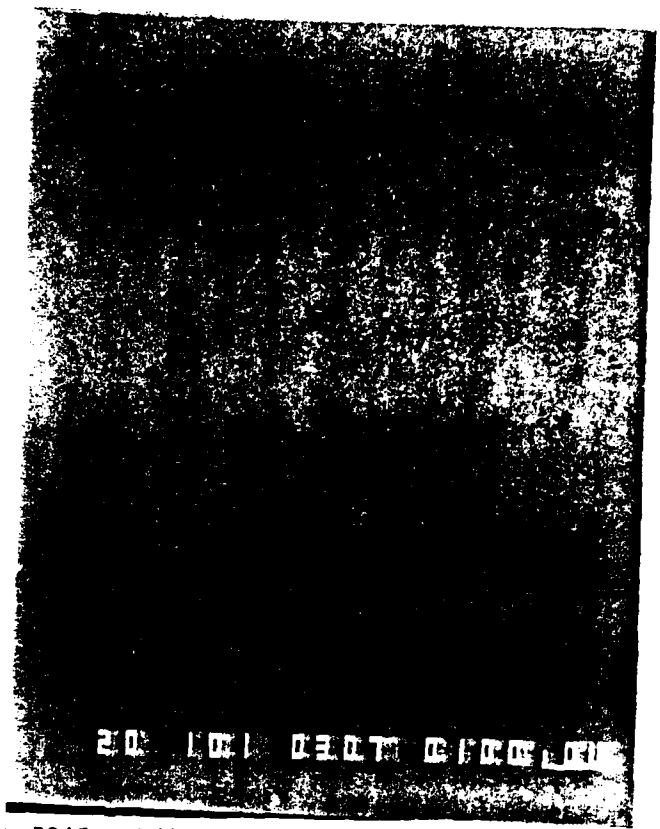
Heat 5256 - Adjacent to the toe and taken from the mid-radius location - Transverse 100X



Heat 5256 - Adjacent to the toe and taken from the centerline location - Transverse 100X



t 5245 - Adjacent to the toe and taken from the O.D. location - Transverse 100X



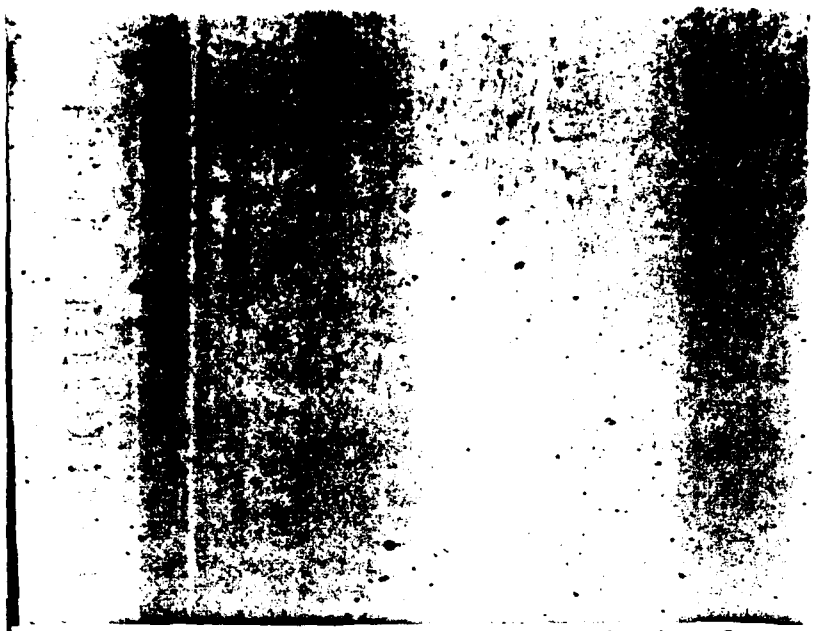
Heat 5245 - Adjacent to the toe and taken from the mid-radius location - Transverse 100X

at 5245 - Adjacent to the toe and taken from the centerline location - Transverse 100X

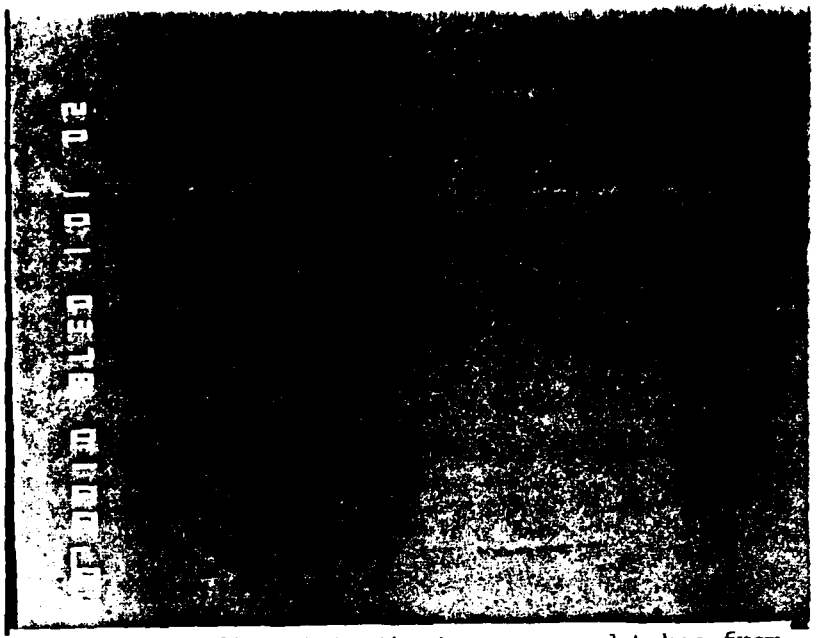
Heat 5245 - Adjacent to the top crop and taken from the centerline location - Transverse 100X

Heat 5245 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X

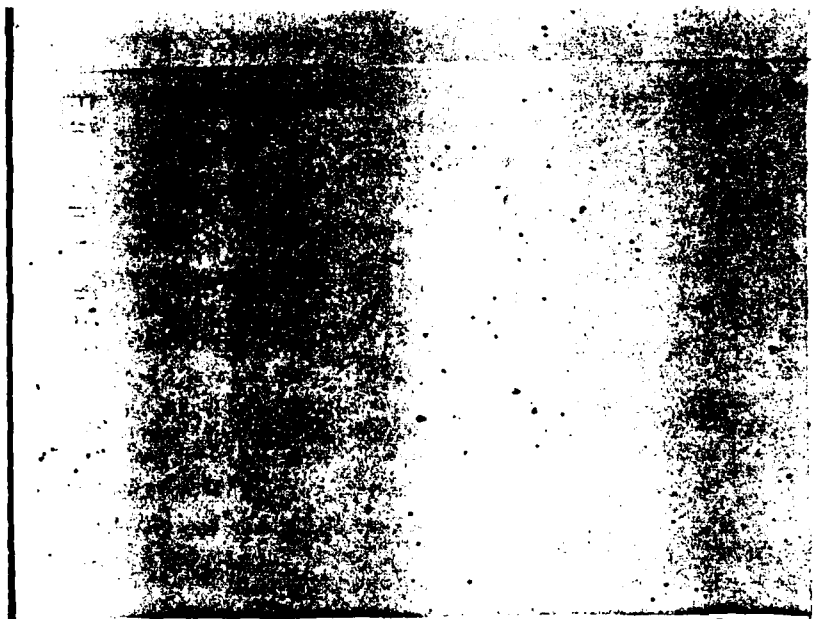
Heat 5245 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X



Heat 5251 - Adjacent to the top crop and taken from the centerline location - Transverse 100X



Heat 5251 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X



Heat 5251 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X

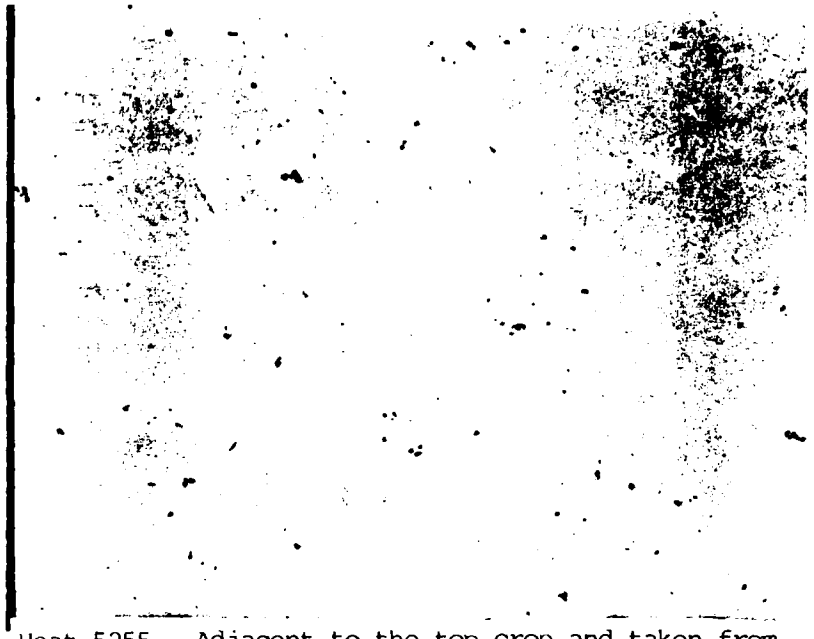
Heat 5251 - Adjacent to the toe and taken from the O.D. location - Transverse 100X

5251 MA C

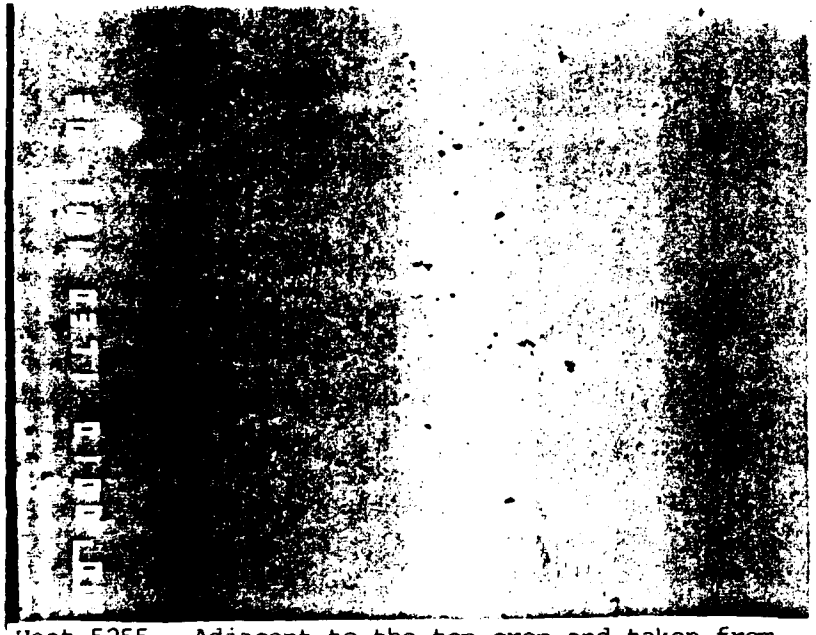
Heat 5251 - Adjacent to the toe and taken from the mid-radius location - Transverse 100X

5251 MA M

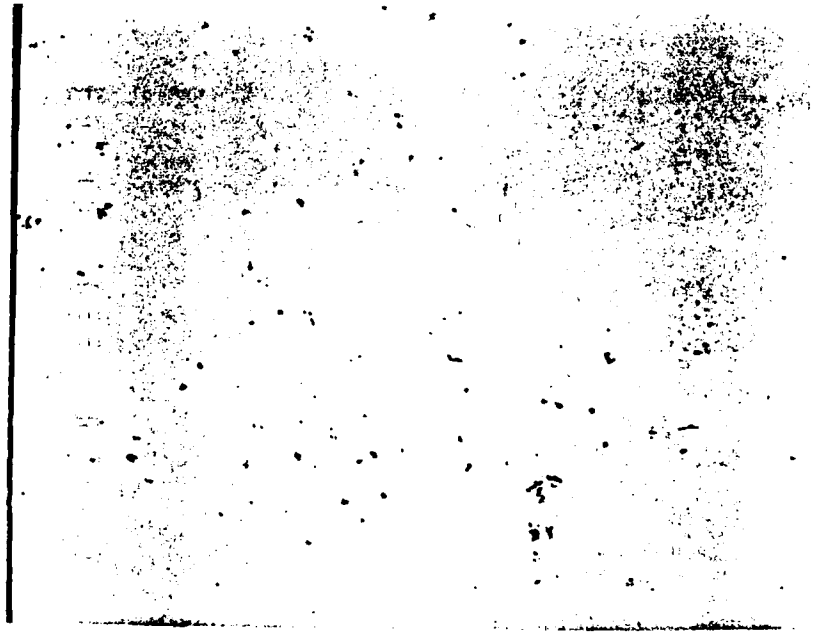
Heat 5251 - Adjacent to the toe and taken from the centerline location - Transverse 100X



Heat 5255 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X



Heat 5255 - Adjacent to the top crop and taken from the centerline location - Transverse 100X



Heat 5255 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X

Heat 5255 - Adjacent to the toe and taken from the O.D. location - Transverse 100X

Heat 5255 - Adjacent to the toe and taken from the mid-radius location - Transverse 100X

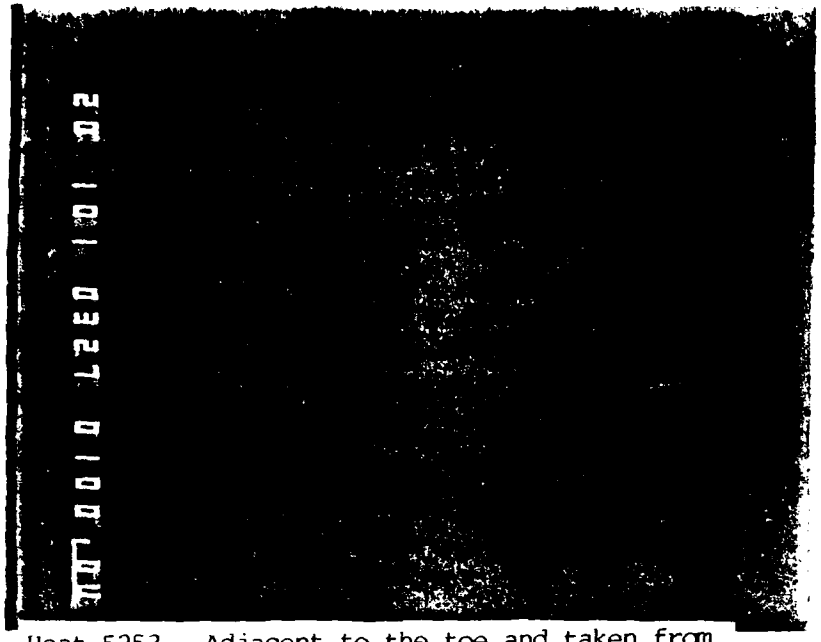
Heat 5255 - Adjacent to the toe and taken from the centerline location - Transverse 100X

Heat 5253 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X

Heat 5253 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X

5253C11 71

Heat 5253 - Adjacent to the top crop and taken from the centerline location - Transverse 100X



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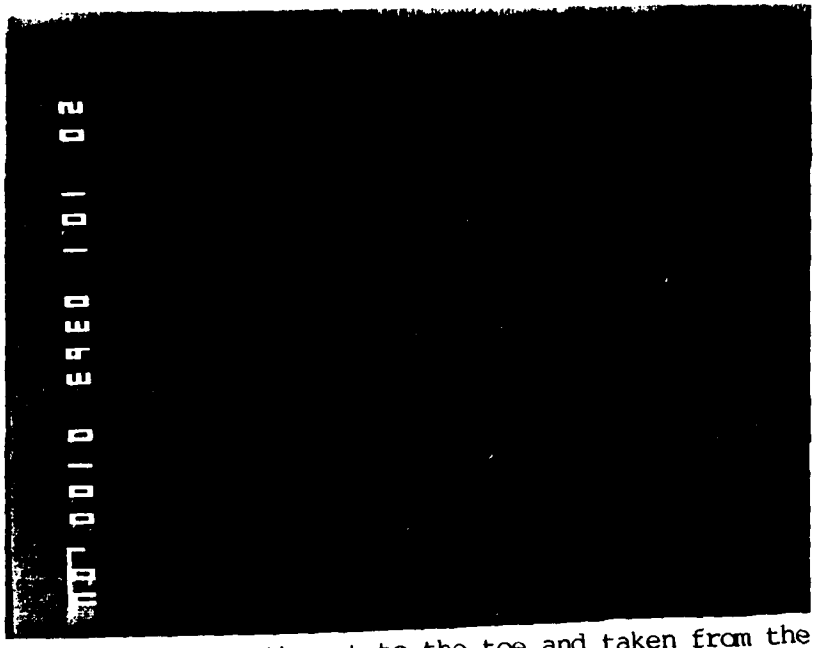

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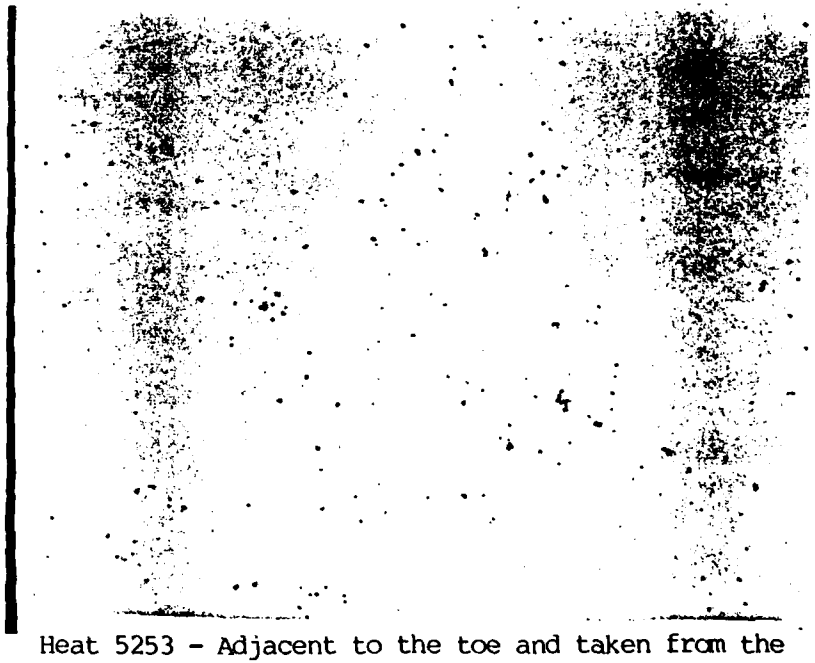


Heat 5253 - Adjacent to the toe and taken from the mid-radius location - Transverse 100X

Heat 5130 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X

5130 A22 O

Heat 5130 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X

5130 A22 M

Heat 5130 - Adjacent to the top crop and taken from the centerline location - Transverse 100X

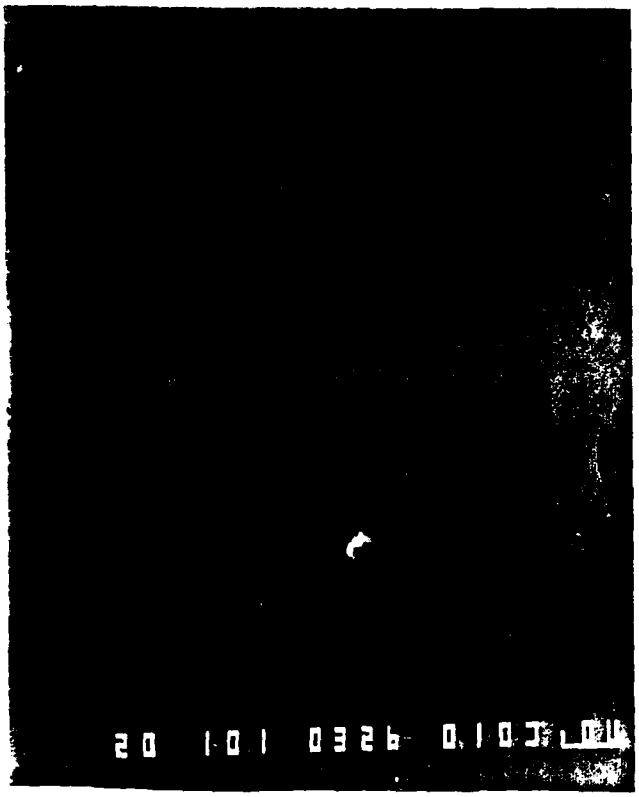
Heat 5056 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X

5056 HE O

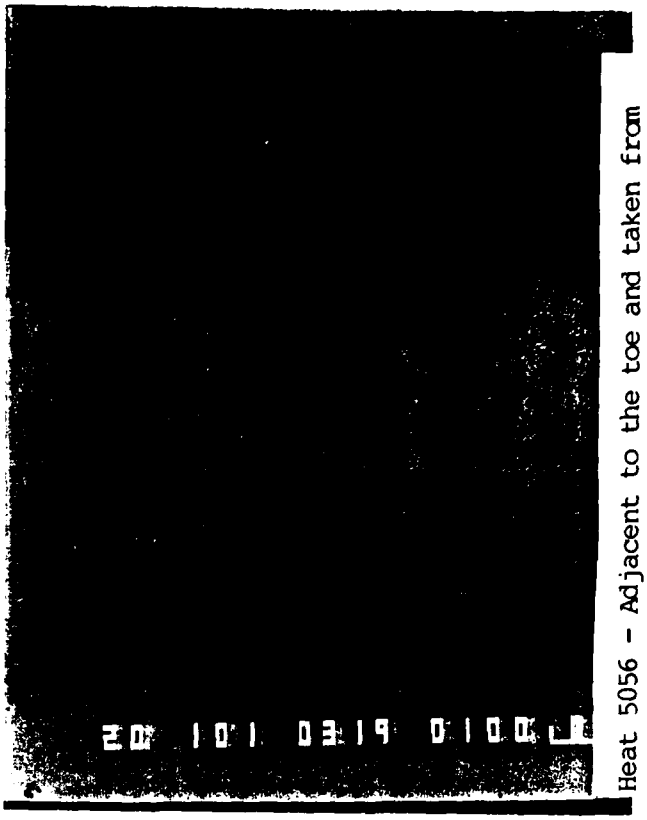
Heat 5056 - Adjacent to the top crop and taken from the mid-radius location - Transverse 100X

5056 MR M

Heat 5056 - Adjacent to the top crop and taken from the centerline location - Transverse 100X



eat 5056 - Adjacent to the toe and taken from the O.D. Transverse 100X



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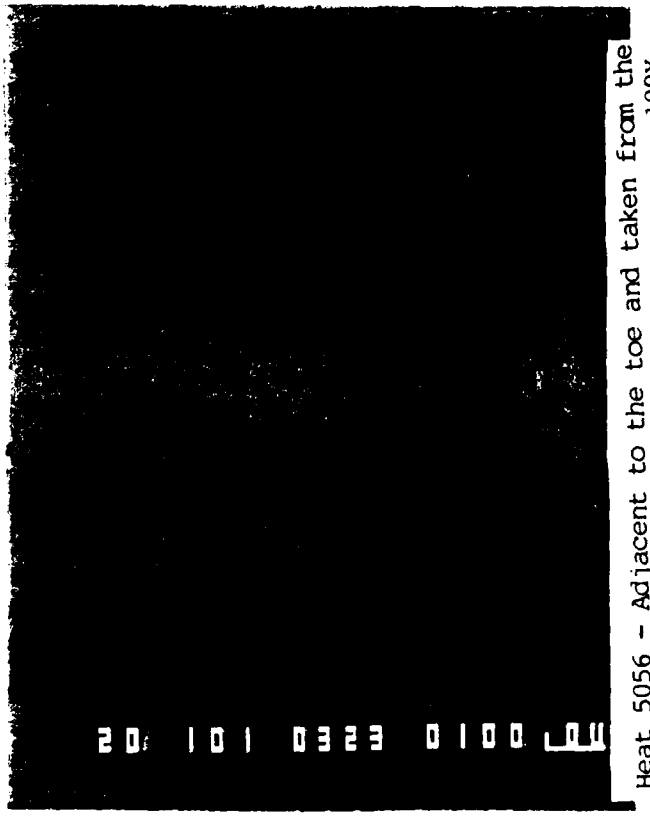
5056 Hll 7/7



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Heat 5072 - Adjacent to the top crop and taken from the O.D. location - Transverse 100X

5072A2 C

Heat 5072 - Adjacent to the top crop and taken from the centerline location - Transverse 100X

Heat 5072 - Adjacent to the toe and taken from the O.D. location - Transverse 100X

Heat 5072 - Adjacent to the toe and taken from the mid-radius location - Transverse 100X

Heat 5072 - Adjacent to the toe and taken from the centerline location - Transverse 100X

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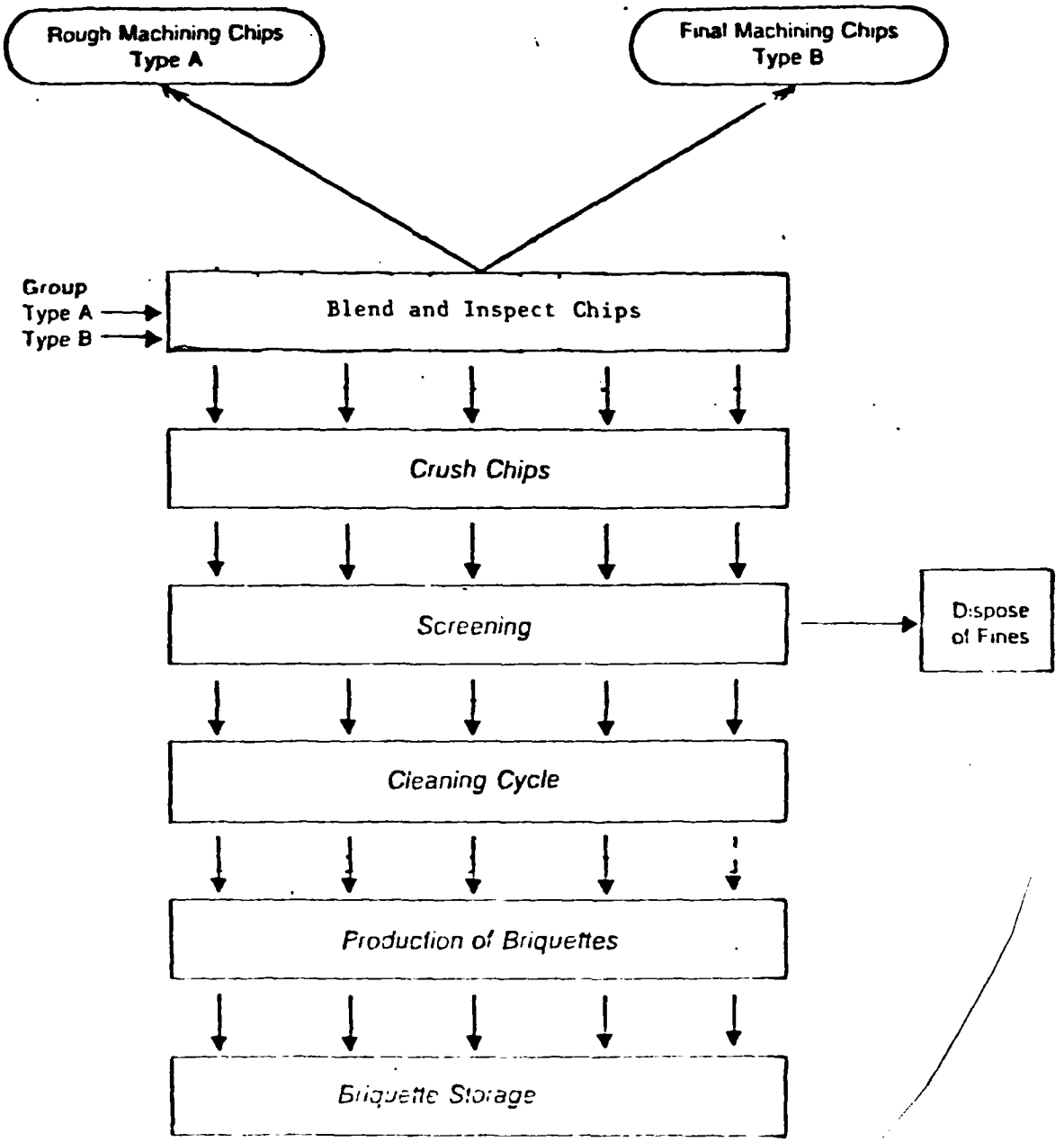
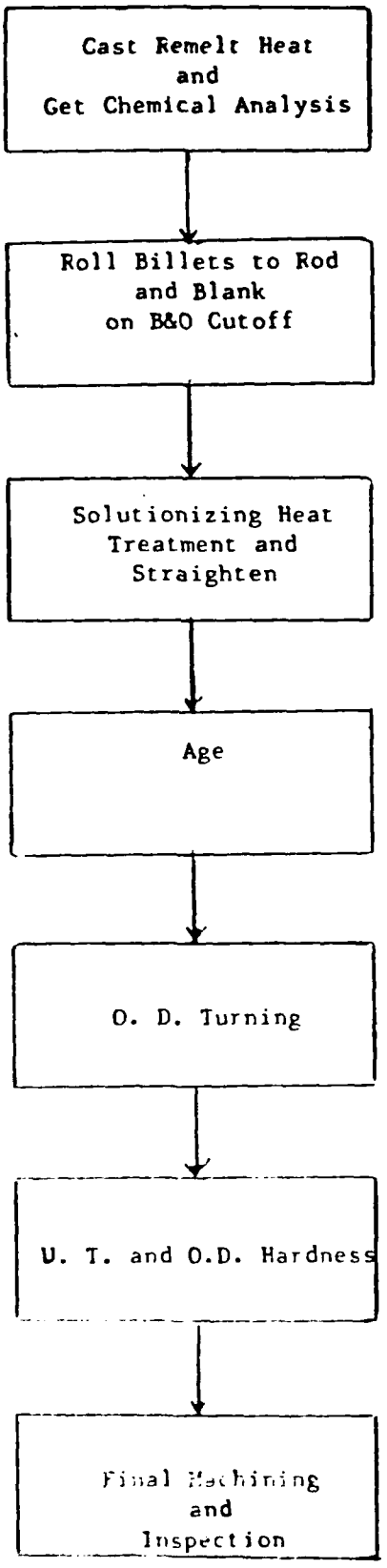
APPENDIX B

CHIP PROCESSING PROCEDURES

Briquetting



PROCESSING 7% CHIP BRIQUETTE THROUGH FINAL CORES



PROPOSED PROCESSING PROCEDURE No. 1

ENTERING, EXITING AND WORKING SAFELY IN THE PRODUCTION AREA

1.0 PURPOSE:

To ensure employee awareness of and compliance with the safety rules and regulations, as defined in the TNS Handbook, General Safety Rules and applicable operating procedures and to ensure employee awareness of the equipment available for personal protection.

2.0 ENTERING AND EXITING PRODUCTION AREA:

2.1 All production employees will:

2.1.1 Collect the TLD badge from the guard station in exchange for your identification badge.

2.1.2 Enter the production area through the clean/controlled locker room only.

2.1.3 Remove personal clothing in the clean locker room and store clothes in assigned locker. Personal clothing, tools, etc., are not permitted beyond the clean locker room.

2.1.4 Put on Company-furnished clothing and yellow toed safety shoes.

2.1.5 Put on safety glasses with side shields prior to entering the production area.

2.1.6 Attach your TLD badge to the collar or vest pocket of your Company-issued clothing.

2.2 Existing the production area all production employees will:

2.2.1 Exit into the controller locker room only.

2.2.2 Remove Company-furnished cotton clothing and yellow-toed safety shoes and store in assigned locker. These clothes are not permitted in the clean area.

2.2.3 All employees must wash before smoking, eating, or leaving the locker room.

2.2.4 At the end of the shift, it is mandatory that the employee shower prior to leaving the clean locker room. This shall include washing hair.

2.2.5 Return the TLD badge to the guard station at the end of the shift to receive your identification badge.

2.2.6 A radiation monitor is located outside the locker room doors for the employee to check his/her body or clothing before leaving the building.

2.2.7 All items must be checked through Health and Safety prior to leaving the plant or production area.

3.0 WORKING SAFELY IN PRODUCTION AREAS:

3.1 Safety glasses with side shields, ear protection (where applicable), TLD badges, safety shoes and Company-furnished clothing are to be worn at all times in the production area by operations employees. Office personnel and visitors will wear safety glasses with side shields, TLD badges, lab coats, shoe covers and ear protection (where applicable).

3.1.1 Gloves shall be worn in accordance with P.O.P. No. 154.

3.2 Food, drink, chewing gum, medicine (aspirin, cold tablets, etc.) and tobacco are not allowed in the production area or controlled locker rooms.

3.3 Long hair must be kept up at all times when working around moving equipment.

3.4 Each employee is responsible for keeping the production area clean during the course of the workday. All work areas will be thoroughly cleaned at the end of each shift, under the direction of the shift foreman.

3.5 Face shields, gloves and aprons must be worn when working with acids.

3.6 Welding hoods, gloves and respiratory protection (if applicable) must be worn when welding.

3.7 Respirators must be worn in areas designated by Health and Safety or at the foreman's discretion.

3.7.1 There shall be no facial hair at the seal area of the respirator. (This includes full face respirators.)

AD-A165 790 PRODUCTION PROVE-OUT OF A PROCESS FOR REMELTING DEPLETED URANIUM MACHININ..(U) AEROJET ORDNANCE CO JONESBORO TN HEAVY METALS DIV C G SKILLERN ET AL. 2/2

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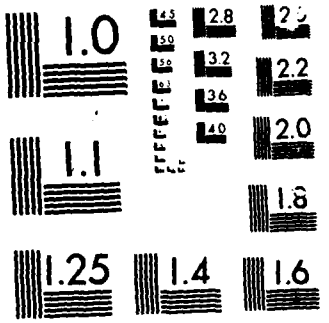

3.8 All accidents, regardless of severity, are to be reported to the foreman, as defined in P.O.P. No. 16, and appropriate treatment obtained. Failure to do so will result in disciplinary action.

3.9 A required urine sample will be taken every two weeks, or as defined by Health and Safety, from all TNS employes before you can clock in to start work. No exceptions will be made.

3.10 Length of sleeves of uniforms will be determined by the Health and Safety Department, according to the type equipment the employee is operating. Rings, necklaces, and loose jewelry shall not be worn by personnel operating or repairing equipment.

3.10.1 TNS-issued uniforms shall not be modified or marked on without approval of the Health and Safety Department.

3.10.2 Disposable protective clothing is available for specific jobs that might require additional protection.

3.10.3 Old gloves, worn-out uniforms, shoes, etc. must be turned in to the foreman when new ones are issued.

3.11 All hats worn on controlled side shall not be taken out of the controlled area unless released by Health and Safety.

4.0 SAFETY HIGHLIGHTS:

4.1 PURPOSE: To present basic safety criteria to all TNS employees.

4.2 Know and carry out your safety responsibilities to protect yourself and all other personnel, as defined in the TNS Handbook, General Safety Rules and P.O.P.s.

4.3 Report all safety hazards to your supervisor.

4.4 Wear protective equipment as provided and required.

4.5 Discuss any safety or health problem with your supervisor. He is your link with company management.

4.6 Always report a job-related injury or illness, no matter how slight.

4.6.1 All incidents and near-misses shall be reported.

4.6.2 Employees shall not be permitted into the production area with open wounds unless they are covered and approved by the Health and Safety Department.

4.7 Never operate faulty equipment; contact your supervisor.

4.8 Never circumvent any safety devices.

4.9 Keep your work area free of safety hazards.

4.10 Remember that horseplay and running are not allowed.

4.11 Think before you act. Make TNS a safe place to work.

PROPOSED PROCESSING PROCEDURE No. 2

CHIP CRUSHER

1.0 PURPOSE:

This procedure provides steps for operating the chip crusher to break up machine turnings into smaller chips for pressing into recyclable briquettes.

2.0 SAFETY:

2.1 Comply with P.P.P. #1.

2.2 Respirator required when adding chips to crusher.

2.3 Slide gate must be closed when adding chips to hopper.

2.4 Gloves required to handle chips.

2.5 Fire extinguishing equipment must be available.

2.6 Verify the magnahelic gage on the vent system reads in the proper range.

3.0 EQUIPMENT:

3.1 Chip crusher.

3.2 30 gallon drum.

3.3 Crane and drum lifting equipment.

3.4 Rake.

4.0 PROCEDURE:

4.1 Verify the magnahelic gage on the vent system reads in the proper range.

4.2 Energize equipment with breaker box on the left. Push "Start".

4.3 Allow equipment to warm up at least two minutes.

4.4 Place 30 gallon drum in bin under hopper to receive crushed chips. Drum must be half full of water.

4.5 Pump water out of a drum of chips.

4.5.1 Chips must be covered with water when being stored.

4.6 Attach drum lift and crane to drum.

4.7 Lift drum and position at hopper.

4.8 Close hopper slide gate.

4.9 Fill hopper with turnings (chips).

4.10 Move drum away.

4.11 Open slide gate and let turnings fall into crusher.

4.12 Close slide gate.

4.13 Perform crushing operation. Chips will fall from exit port below crusher and into 30-gallon drum located beneath crushing unit. This 30-gallon drum will contain water to ensure chip coverage.

4.14 Upon completion of crushing operation, remove drum containing crushed chips. Care must be taken to clean up any stray chips/residue.

4.15 Move drum containing crushed chips to storage.

PROPOSED PROCESSING PROCEDURE No. 3

CHIP CLEANING AND PICKLING

1.0 PURPOSE:

This procedure provides the steps to clean residual coolant from the lathe turnings and the pickling process to remove excess oxides from the chip surfaces.

2.0 SAFETY:

2.1 Comply with P.P.P. #1.
2.2 Gloves required to handle chips.
2.3 Face shield and liquid repellent coat must be worn while pickling.
2.4 Operator must not stand under the crane during operations.

3.0 EQUIPMENT:

3.1 55 gallon drums of 5% Trim-Rinse solution and rinse water.
3.2 New Holland spin dryer with 150 lb. capacity.
3.3 Stainless steel mesh baskets for cleaning and pickling.
3.4 Overhead crane and drum lifting equipment.

4.0 PROCEDURE:

4.1 Drain coolant from crushed chips.
4.2 Sieve the crushed chips over a 12 mesh screen mounted on a barrel of coolant.
4.3 Place sieved chips in a stainless steel mesh basket.
4.4 Load the basket of chips into the detergent wash for three minutes with the crane. Agitate with an up and down motion of the crane.
4.5 Drain the basket by holding over the detergent barrel for three minutes.
4.6 Load the basket of chips into the water rinse for three minutes with the crane. Agitate with an up and down motion of the crane.
4.7 Drain the basket by holding over the rinse barrel for two minutes.
4.8 Load the spin dryer and run for 10 minutes with no heat.
4.9 Transfer the basket of chips to a wheeled dolly and move it to the nitric acid pickling area.
4.10 Dip the basket of chips into the rinse tank using the pickling crane.
4.11 Drain the basket of chips over the rinse tank and load into the nitric acid solution pickling tank.

4.12 Hold the basket in the nitric acid tank for a period of 10-40 minutes until chips are clean.
4.13 Remove the basket of chips from the acid bath and place in the rinse tank, agitating with an up and down motion using the crane.
4.14 Drain the basket of chips by holding it over the rinse tank for two minutes.
4.15 Transport the basket of chips to the spin dryer and dry for ten minutes without heat.
4.16 Place the chips in a 20 gallon drum with dry ice.

PROPOSED PROCESSING PROCEDURE No. 4

CHIP BRIQUETTING PRESS

1.0 PURPOSE:

This procedure provides steps for operation of the chip recycling press to compact machining chips into a briquette to use as remelt scrap.

2.0 SAFETY:

2.1 Comply with P.P.P. #1.

2.2 Gloves required to handle chips.

3.0 EQUIPMENT:

3.1 300 ton Dake press.

3.2 Dies

3.3 Rams

4.0 PROCEDURE:

4.1 Chips should be chemically cleaned before pressing.

4.2 Energize press with breaker box and start button on left.

4.3 Load chips into die.

4.4 Position die under ram.

4.5 Lower ram by pushing handle on right down.

4.6 Verify ram aligns with die. Stop and re-position die if necessary.

4.7 Periodically raise ram and twist die.

4.8 Continue pressing until pressure on gage (upper right) reads approximately 300 tons.

4.9 Raise ram by raising handle on right.

4.10 Invert die and place under ram.

4.11 Lower ram until pressed briquette breaks loose.

4.12 Raise ram, remove die and take out pressed briquette.

4.13 Examine briquettes for loose flaking hips, which will be cause to scrap the briquettes.

4.14 Briquettes will be transferred to 20 gallon covered storage containers on wood pallets. No more than 40 briquettes will be stored in each container.

4.15 Three one-pound slabs of dry ice will be added to the storage containers every other day until remelted.

PROPOSED PROCESSING PROCEDURE No. 5

CASTING OF BILLET FOR M833 PENETRATORS

1.0 SCOPE:

1.1 PURPOSE - This ordnance engineering doument is a process specification for the casting of depleted derbies and/or reprocessed materials into billets containing 3/4 weight percent (w/o) titanium.

1.2 Application - This document covers the processing of solid uranium metal into billets including the preparation of molds and crucibles.

2.0 MATERIALS AND EQUIPMENT:

2.1 Materials required:

2.1.1 High purity titanium
2.1.2 Depleted Uranium (various forms, derbies, mics. scrap)
2.1.3 Ethyl alcohol (commercial grade)
2.1.4 Graphite coating slurries and paints

2.2 Equipment:

2.2.1 Graphite mold and crucible cleaning and coating equipment.
2.2.2 Vacuum remelt furnaces with vacuum pumps and power supplies.

3.0 SAFETY AND HANDLING PRECAUTIONS:

3.1 Safety glasses/side shields, shoes and company furnished clothing are required throughout operation.

3.2 Protective apron, face shield, hot gloves and respirators when required.

3.3 Ventilation required where specified.

3.4 TLD badges are required.

4.0 PROCEDURE FOR BILLET PRODUCTION:

4.1 Graphite mold preparation:

4.1.1 Select mold components from graphite inventories.

4.1.2 If previously used, remove all traces of previous coating using cleaning equipment and ventilation provided.

4.1.3 Apply appropriate protective coating to interior of mold sleeves.

4.1.3.1 Inspect interior of mold sleeves. If any unacceptable runs or curtains are present, reclean.

4.1.4 Bake coated mold sleeves.

4.1.5 Place baked out mold sleeves on base and assemble diverter and top plugs.

4.1.5.1 Use ceramic paste to seal top plugs in place.

4.1.5.2 Paint proper surfaces of graphite mold assembly with appropriate coating slurry and air dry as required.

4.1.5.3 Complete assembly by wrapping paper portion with layer of insulation materials.

4.2 Crucible preparation:

4.2.1 Select new or used graphite crucible, stirring rod, lid and pour plug.

4.2.2 Remove old coating from previously used components.

4.2.3 Apply one to two coats of slurry to the inside of the crucible, lid and pour plug. Coat the outside of the stir rod.

4.3 Furnace assembly:

4.3.1 Place crucible in induction coil and seal in bottom plug.

4.3.2 Place mold assembly into mold tank. Assembly may consist of up to 12 mold sleeves.

4.3.3 Inspect above units (steps 4.3.1 and 4.3.2) for completeness and integrity of coating. Repair or replace if necessary.

4.3.4 Place pre-weighed uranium and alloy materials into crucible, recording each item charged on the casting charge (sheet no 1).

4.3.4.1 Any combination of traceable derbies and remelt materials may be used.

4.3.4.2 Add sufficient titanium to produce Stoichiometric billet levels of 0.74 $\pm$ 0.05 w/o Ti alloy.

4.3.4.3 Chip briquettes will be loaded on top of the derbies in the crucible.

4.3.5 Clean and grease all "O" rings, mating flanges and gaskets - close furnace.

4.3.6 Place furnace control in vacuum open position and start at least one vacuum pump. Record on billet traveler (sheet no. 3).

4.3.7 When furnace reaches the necessary negative pressure, blank off furnace and observe pressure changes. Record these changes on billet traveler (sheet no. 3). Reopen valves.

4.3.8 When the appropriate negative pressure is obtained, apply crucible and mold power.

4.3.9 Record all information concerning start up and run on the casting record sheet (sheet no. 2). This includes time, furnace vacuum, crucible power, mold power and any remarks about process that are unusual.

4.3.10 After the melt has pooled, reduce the input power.

4.3.11 When the specified pour temperature or time is reached and the melt stirred, shut down the power and pour immediately.

4.3.11.1 Record the time and all pertinent data as above including any comments about pour conditions, such as rapid changes in pressure, splattering in the mold, etc. on the casting run record.

4.3.11.2 Allow the vacuum to stabilize within the furnace and record on the casting run record.

4.3.12 Blank furnace off and proceed with cooling bleed back process.

4.3.12.1 Ensure that billet traveler is complete and accurate.

4.3.13 Remove casting assembly from furnace. Remove the graphite mold and sample all appropriate billets top and bottom for chemistry per MIL-C-63422A (Top samples are obtained after cropping).

4.3.13.1 Metal stamp number all billets per traceability specification on PCD 830029-833.

4.3.13.2 Store sampled billets in designated area until an OK chemistry is returned.

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APPENDIX C

PREPARATION OF DU MACHINING CHIPS

BY

SODIUM CARBONATE PICKLING

FOR

VACUUM INDUCTION REMELTING

SODIUM CARBONATE PICKLING

INTRODUCTION

Nitric acid solutions are used to remove loose scaly uranium oxides at various stages of penetrator production. It removes the oxides formed during rolling along with the residual salts from the rolling mill salt bath. It is also very effective for cleaning the heavy oxide formed on the ingot top (top crops) after casting in the VIR (Vacuum Induction Remelt) furnace. While nitric acid is an effective way to clean in process uranium in process, it has two major drawbacks which are discussed in detail below.

The first drawback associated with nitric acid pickling is the total nitrate emission discharged in the facility's waste water. Nitric acid is highly water soluble and cannot be removed from waste water using the current AHMC facility. The Tennessee Department of Water Quality under the EPA has limited the AHMC daily nitrate emission in the water discharge to 50 pounds per day. AHMC would be precariously close or, in fact, over this maximum level at mobilization production rates.

The second drawback associated with nitric acid pickling is the emission of toxic nitrous oxide, a by-product of the nitric acid pickling process. Safe handling of nitrous oxide requires an extensive ventilation system along with a scrubber to remove the nitrous oxide from the exhaust gases. The scrubber forms dilute nitric acid from the nitrous oxides which are somewhat difficult to handle in waste water streams.

The introduction of chip recycling to AHMC production, if it required HNO_3 pickling of the chips, would raise the amount of nitrate discharge very close to the regulated maximum allowable. Accordingly, we must find an alternative to our present process.

There are at least two viable approaches to overcome these environmental problems. The first is to use anaerobic bacteria to break down the nitrates. The use of anaerobic bacteria would require a facility with stringent process controls, and even after the nitrates were decomposed, the end products would still have to be processed through the traditional waste treatment facility. Although this solution is practiced with success in industry, it is desirable to identify a simpler solution.

A second approach is to develop an alternative cleaning (pickling) media, which does not have the same environmental difficulties. Major criteria for this alternative cleaning media would be effective oxide removal on chips and easily treatable waste products.

Solutions of sodium carbonate and hydrogen peroxide were found to provide a viable alternative to nitric acid. Saturated sodium carbonate solutions with 1-1.5% hydrogen peroxide additions can remove the various forms of uranium oxide (U_3O_8, UO_2, etc.) from machining chips. The liquid pickling by-product, uranyl carbonate, is readily precipitated out by the addition of lime and NaOH. Carbonates remaining in solution do not have the environmental impact of nitrates and no limit is currently set on them.

Contrary to HNO_3 pickling, the gaseous by-products of sodium carbonate pickling are predominantly oxygen with some hydrogen. Since both gases are released slowly, only a simple ventilation system with demisting will be required. Furthermore, since the gases are not toxic as is the NOx released in HNO_3 pickling, the need for elaborate ventilation and scrubbing is eliminated.

For these reasons, AHMC proposed to the Army that an effort be added to the Chip Recycle MM&T, contract DAAK10-83-C-0248, with the objective of determining whether Na_2CO_3/H_2O_2 could form the base for an effective DU machining chip cleaning system. The work was divided into four tasks as discussed below.

Task A. Kinetics of Oxide Removal from Solid Blanks.

Scoping experiments showed that the oxide removal could be followed using weight loss as an experimental measure. Rolled 1.18" diameter rod was cut into 1-inch long sections. These were cleaned in a 50-50 HNO_3/water solution at 25°C for about 20 minutes or until they were silvery in color (indicating little, if any, residual oxide). These cleaned samples were then oxidized in an air oven for various times at 150, 315 and 427°F to produce oxide films representative of machining chips with light, moderate and severe oxide films.

An estimate was made of the oxide film thickness on these samples using metallography. A technique was developed to measure the thickness of the DU-3/4 wt.% Ti oxide formed. To insure edge retention during polishing, samples were electrolytically copper plated using a DC power source. The plating solution was a copper sulphate-water solution (250g/l) with a 10% addition of dilute sulfuric acid. Optimum plating power settings were 15 volts and 1/2 amp. The copper plate allowed normal metallographic polishing of the samples without destroying or distorting the oxide edge.

Figure 1 shows a 450X photograph of a sample oxidized at 204°C for 20 minutes. The thin dark line is the layer of uranium oxide formed at the sample's edge. Measurement on the metallograph revealed an 0.5 micron thick oxide layer. Several other samples were plated and their oxide layers were also 0.5 microns thick. A number of samples were oxidized at different times and temperatures to simulate the oxide layer found on chips. A listing of the oxidizing times, temperatures, and oxide thicknesses are shown in Table 1. Although the measurements show wide scatter, it can be seen that oxide thicknesses in the range of 1 to 20 μ were formed.

Weight Loss Rate Studies

The oxidized bar samples (20 for each experiment) were immersed for times from 1 to 20 minutes in a solution saturated with Na_2CO_3

and containing one of two levels of H_2O_2, 0.6 or 1.2%. In addition, samples were immersed in an 0.6% H_2O_2 saturated Na_2CO_3 solution at temperatures of 25, 40, and 55°C. Specimen weight loss as a function of time was determined for each of these five conditions.

Hydrogen Peroxide Decomposition

Besides looking at oxide removal rates, it was important to determine the relative life of the hydrogen peroxide in the sodium carbonate pickling bath. A colormetric technique was used to determine the hydrogen peroxide concentrations in the bath. Key to the technique was the titanium oxide and ammonium sulfate solution which reacted with the unspent hydrogen peroxide. After two minutes, the color change could be measured on the photospectrometer (light spectroscopy) as absorbance at a wavelength of 428 MU. Absorbance and the unspent hydrogen peroxide were correlated in a linear relationship. Previous work showed a relationship like the following:

$$Y = A + B\ (X)$$

A = 0.0037
B = 0.78681
X = Absorbance (%)
Y = Hydrogen Peroxide Concentration (Mg/25cc H_2O_2)

The actual spectrographic samples were made by extracting 25 mls of pickling solution from the sodium carbonate bath. Five mls of the titanium oxide solution were added to the 25 mls of pickling solution and held for two minutes. After two minutes, 10 mls of the combined solutions were added to an absorbance cell and the solution's absorbance was read on the photospectrometer.

The H_2O_2 bath stability was determined by immersing oxidized bar samples for 10 minute time periods in the bath. After 10 minutes, a new oxidized bar was put into the bath. Both temperature

and initial hydrogen peroxide concentration were varied and the residual H_2O_2 concentration was measured periodically for a total time of 60 minutes.

Task B. Removal of Uranium Oxide From DU-3/4 wt.% Ti Machining Chips

The rate of weight loss for machining chips was determined at two temperatures, 20^{o} and $40^{o}C$ at a nominal 1.15% H_2O_2. These conditions were chosen on the basis of the Task A results. In addition, hydrogen peroxide decomposition curves were determined at temperatures of 25^{o} and $40^{o}C$. The hydrogen peroxide concentration of 1.15% was optimal due to its rapid and thorough removal of the uranium oxide.

Care was taken in this task to ensure that the ratio of chip surface area to pickle solution volume was relatively the same was used in the Task A studies. It was felt that maintenance of constant area/volume ratios would ensure that H_2O_2 decomposition rates would be essentially the same. The average surface area of chips was determined qualitatively using optical microscopy. Chips were determined to have 3.8 cm^2/gm surface on the average. Using this average chip surface area, a constant surface area to volume ratio of .2226 square centimeters per milliliter of pickling solution was used.

Task C. Sodium Carbonate Pickling of a Production Scale Batch of Machining Chips.

All chips in this portion of the Chip Recycling MM&T were collected from the Central Chip Collection System (CCCS) and heat identity of the chips was impossible. Random barrels of chips from the CCCS were selected from the storage backlog. Each barrel contained about 125 pounds of lathe turnings (machining chips) submerged under coolant to preclude a fire hazard. Five barrels of chips were sent from the Coolant Recovery Area to the Chip Processing Area. Each barrel contained a blend of coarse O.D. turning chips and fine finish turning chips.

Coolant was drained from each of the barrels and the chips were crushed damp in the American Ring Crusher just as in the earlier portion of the MM&T study. After crushing, the chips were sized over a 12 mesh screen to remove chip fines. The chips were then cleaned in a 5% solution of Trim-Rinse for five minutes with an up and down agitation motion. Washed chips were rinsed for five minutes in clean rinse water. The chips were spin dried and were now ready for sodium carbonate pickling.

The production sized batch of chips required a rather make-shift sodium carbonate pickling facility. 55 gallon steel drums were used for both the pickling and rinse solutions. The carbonate cleaning solution was prepared by adding 40 gallons of water to 74 pounds of sodium carbonate and 1-1/2 gallons of 35% concentration hydrogen peroxide. The rinse barrel was prepared by cleaning a rust free 55 gallon barrel and filling it with 40 gallons of clean water.

150 pound batches of chips were pickled for 20 to 30 minutes at 25^{o}C. Three batches of chips were pickled to yield 410 pounds of chips.

After pickling, the chips were rinsed and dried as before. The next day the chips were briquetted. The briquettes were melted as part of Heat 5742 three days later. This heat contained 400 pounds of chips, e.g. 12% of chips.

Task D. VIR Remelting of Heat No. 5742.

Heat number 5742 had the same constituent charge as the other MM&T chip recycling heats. No top crops were used in the heat. The scrap charge was made of mill scrap and large caliber scrap, rejected blanks and cores. The charge make-up was:

Derby	Scrap	Chips
2066	881	400

Detailed billet chemistries were determined in the same way as on the previous chip heats. A billet was cut into twelve 2-1/2 inch slices; billet slice number 1 was immediately adjacent to the top crop, while slice 12 was the billet bottom. Spectrographic analysis was run on drillings taken from the O.D., mid-radius, and centerline locations of each slice, yielding a total of 36 different chemistries within the billet.

Quantitative microcleanliness analyses were performed on the O.D., mid-radius, and centerline locations of slices 1 and 12. The point intercept inclusion counting technique was used as with the earlier MM&T chip recycling heats.

RESULTS AND DISCUSSION

Task A. Kinetics of Oxide Removal From Solid Blanks.

Weight Loss

The weight loss data are plotted in Figures 2 and 3. These figures show that increasing either temperature or H_2O_2 concentration results in an increase in the rate of initial weight loss and total weight loss. The solid curves given in the figures are logarithmic regression fits to the actual data points. The correlation coefficients for these curves range between 0.9 and 0.95, e.g. a relatively good fit.

Such logarithmic curves may be representative of two competing reactions; the steep initial rate of attack probably represents the dissolution of the various uranium oxides. The later slower rate probably represents the dissolution of uranium metal. This corresponds with experimental observations that the blanks turned a very light gold color (indicative of a very thin oxide) after five to seven minutes. There were only scattered patches of oxide left on the blank at this time.

Hydrogen Peroxide Decomposition

Figure 4 shows the rate of H_2O_2 decomposition at 25°C for cleaning bar samples using initial peroxide concentrations of 0.287, 0.573, and 1.15%. It can be seen that there is an initial rapid decomposition before the first concentration measurement can be made. Nevertheless, after one hour, there was still significant hydrogen peroxide left in the pickling solutions.

Figure 5 shows the hydrogen peroxide decomposition rates at 40°C. All three initial concentrations (0.287, 0.573, and 1.15%) dropped about 60% before the first minute was up. After the first minute, all the decomposition rate curves showed declining unspent hydrogen peroxide concentrations. In fact, the 0.287% initial concentration had no unspent peroxide after an hour. Elevated temperatures accelerated the hydrogen peroxide decomposition rates to the point that the bath life would be very short.

Task B. Removal of Uranium Oxide from DU-3/4 wt.% Ti Machining Chips.

Chip cleaning experiments were conducted using a 1.15% initial hydrogen peroxide concentration at both 25°C and 40°C. Both weight loss and hydrogen peroxide decomposition were determined at each temperature. Figure 6 shows the weight loss curves for 25°C and 40°C bath temperatures. Although a fair amount of scatter exists in the data, it is apparent that chips are behaving in the same way as the solid blanks. Visual observation indicated that the chips were cleaned within 5 minutes at 25°C and 1.15% initial H_2O_2 concentration.

Figure 7, the hydrogen peroxide decomposition rates for solutions used to clean chips at 25°C and 40°C, is qualitatively similar to Figures 4 and 5 with the exception that the high temperature (40°C) decomposition rate is higher when cleaning chips than it was for cleaning blanks. Great care was taken to approximate the same surface area per unit volume both for the blank study and the chip study. In spite of this, it appears that the chip experiments either represented a greater surface

area/unit solution volume or some other uncontrolled variable was present in the experiments.

These experiments indicate that saturated sodium carbonate solution with 1.15% hydrogen peroxide at 25°C will remove oxide from the surface of the chips.

Task C. Sodium Carbonate Pickling of a Production Scale Batch of Machining Chips.

Large scale pickling was done with a 1.15% hydrogen peroxide-saturated sodium carbonate solution at 25°C in a 55 gallon drum. All chips appeared light to medium gold in color after the carbonate pickling for 30 minutes.

Task D. VIR Remelting of Heat No. 5742.

This experimental Heat, #5742, reacted well during melting and casting in the VIR furnace. However, chemical analysis showed that the heat was out of the M833 chemistry specification with respect to iron, 63 ppm.

In order to determine the origin of the high iron, the charge make-up of the heat was carefully examined. The iron levels in the derbies were less than 20 ppm. The mill scrap and large caliber scrap also did not seem to be a source for high iron levels. Therefore, it was suspected that the chips used as part of the charge make-up could possibly be the material supplying high iron levels.

Accordingly, samples of the chips which had been cleaned using the procedures described above were analyzed for iron. Surprisingly, iron levels in the 100-180 ppm level were found. Nonetheless, an iron level of 200 ppm in the chips (11% of the heat charge) would not raise the iron content of the overall heat to above 40 ppm. Hence, the heat's overall iron levels could not be accounted for at the time of this report. Further investigations are continuing.

In spite of the high iron content, Heat #5742 was examined for radial and longitudinal chemistry segregation as originally planned. Figure 8 shows the longitudinal distributions of chemistries for Fe, C, and Si. Both the Fe and Si distributions displayed segregation with high concentrations located at the bottom of the billet. Figures 9 and 10, the radial chemistry distribution of Fe and Si, showed the longitudinal segregation even more prominently. Carbon was not segregated either longitudinally or radially as Figure 11 showed.

Microcleanliness of this heat, as determined by the point intercept inclusion counting technique, was compared with previous chip recycling heats. Figure 12 shows how Heat #5742 compared to the other chip and production heats studied. As can be seen, Heat #5742 was comparable to production billets. As before, all the inclusions appeared to be TiC.

SUMMARY

Experimental results indicated that a nominal 1% H_2O_2-saturated sodium carbonate solution would remove uranyl oxide from the U-3/4 wt.% Ti metal surface. The rate of oxide removal depended both on the hydrogen peroxide concentration and on the bath temperature. Elevated hydrogen peroxide concentrations and bath temperature both increased the oxide removal rate. Experimental results indicated that sodium carbonate pickling was slower than nitric acid pickling.

The iron pickup on chips used in the production heat for this study cannot be explained at this point. For this reason, this pickling approach will not be pursued at this time. It should be pointed out that if further work were to indicate that the iron pickup was atypical, then sodium carbonate/H_2O_2 would be a viable alternative chip cleaning system.

Table 1. List of oxidizing time, temperature, and oxide thicknesses developed for the oxide removal evaluations.

Temp. (°C)	Time	Sample No.	Magnification	Actual Oxide Thickness μ
150	150 minutes		500	1.1125
150	150 minutes		1000	1.9837
315	10 minutes	B1	1000	1.5088
315	10 minutes	B1	650	1.7116
315	10 minutes	B2	430	7.7559
315	10 minutes	B2	650	5.9827
315	10 minutes	B1	650	4.8846
315	15 minutes	P1	350	12.0178
315	15 minutes	P1	800	9.6393
315	20 minutes	A-2	500	2.6975
315	20 minutes	A-2	1000	2.9362
315	20 minutes	A-2	800	2.2876
315	20 minutes	A-1	500	5.5575
427	20 minutes	C2	750	18.0983
427	20 minutes	C1	650	8.3038
427	20 minutes	C2	750	13.8650

Table 2 - Chemical Analysis Results
for Heat #5742

ppm except for Ti (%)

Ti	0.75%
C	52
Fe	63
Al	<8.3
Cd	0.3
Co	1.0
Cr	<7.8
Cu	4
Mg	<2.5
Mn	7.5
Ni	<6.8
Si	68
V	1.1
Zn	7.1

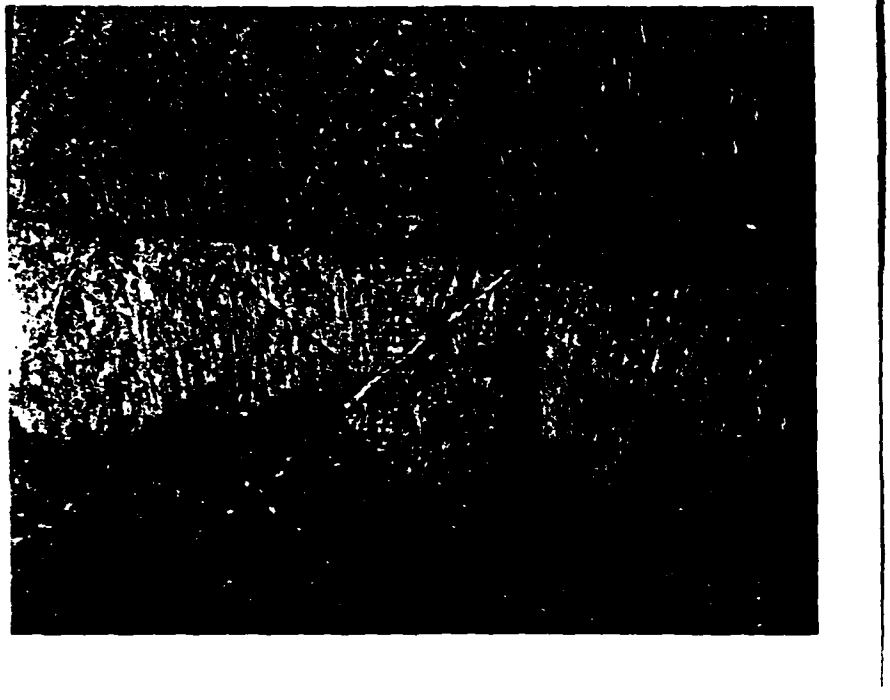

Figure 1. Depleted uranium sample which was oxidized at 204°C for 20 minutes. This 450X microphotograph shows the thin dark uranium oxide layer.

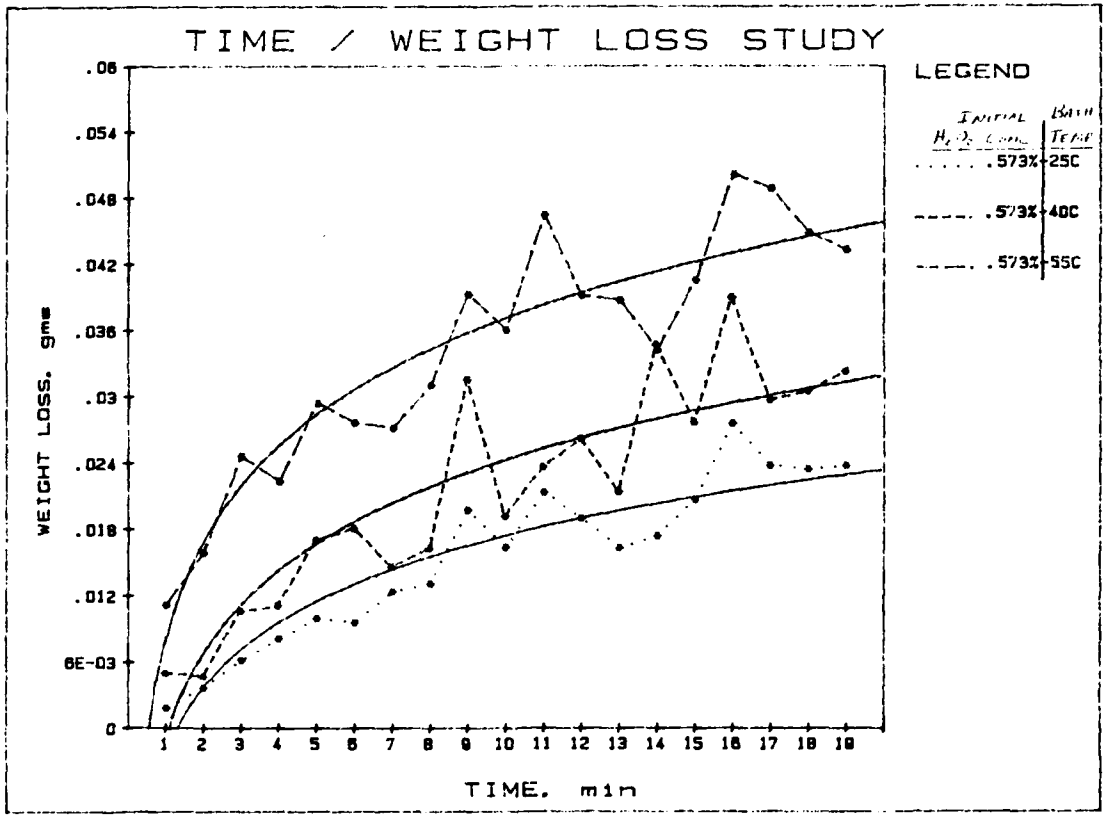

Figure 2. Comparison of the weight loss of oxidized DU rod samples when exposed to constant H_2O_2 concentration at various bath temperatures.

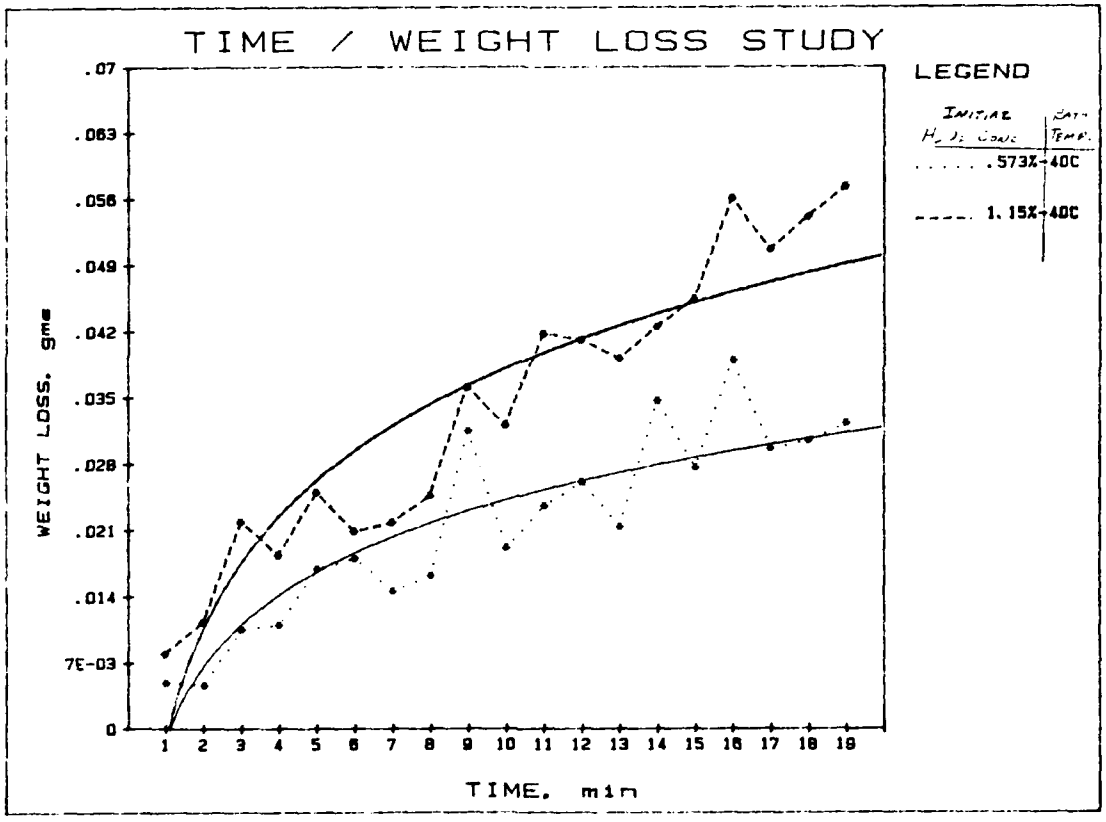

Figure 3. Comparison of the weight loss of oxidized DU rod samples when exposed to various H_2O_2 concentrations at constant Bath temperatures.

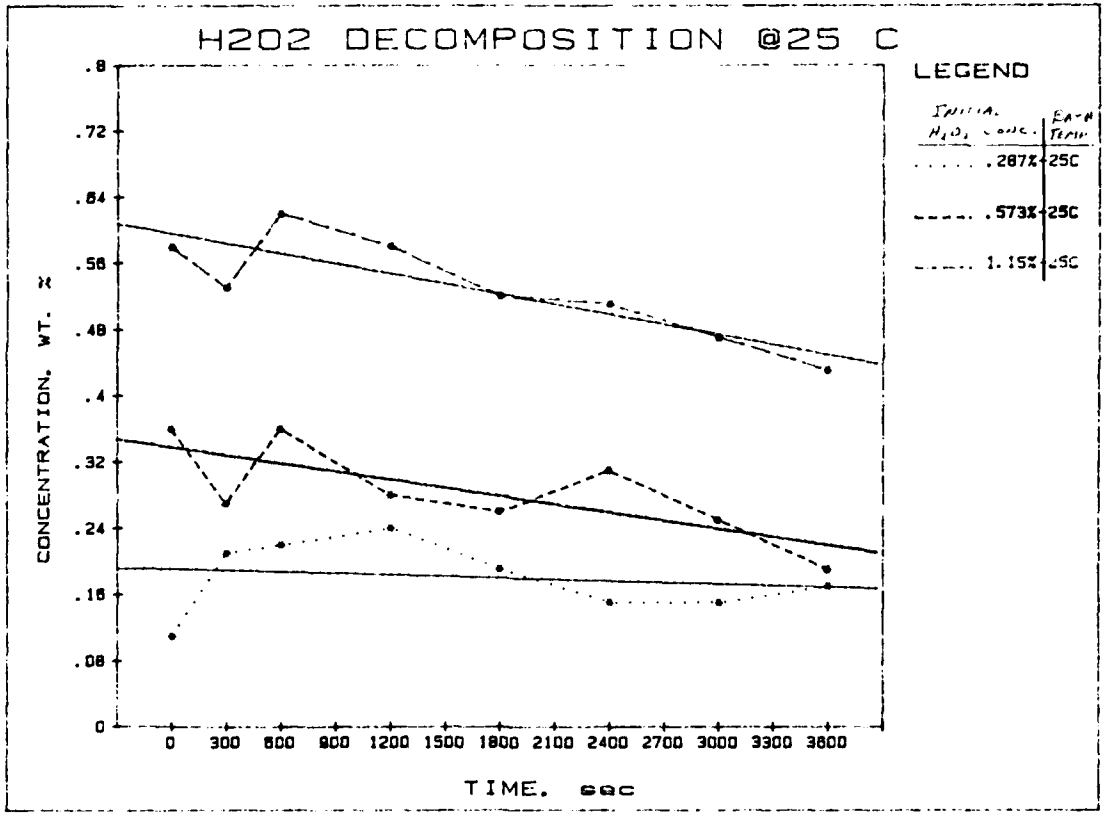

Figure 4. Hydrogen Peroxide Decomposition Rate for removing oxide from DU bar for various initial H_2O_2 concentrations. Bath temperature 25°C.

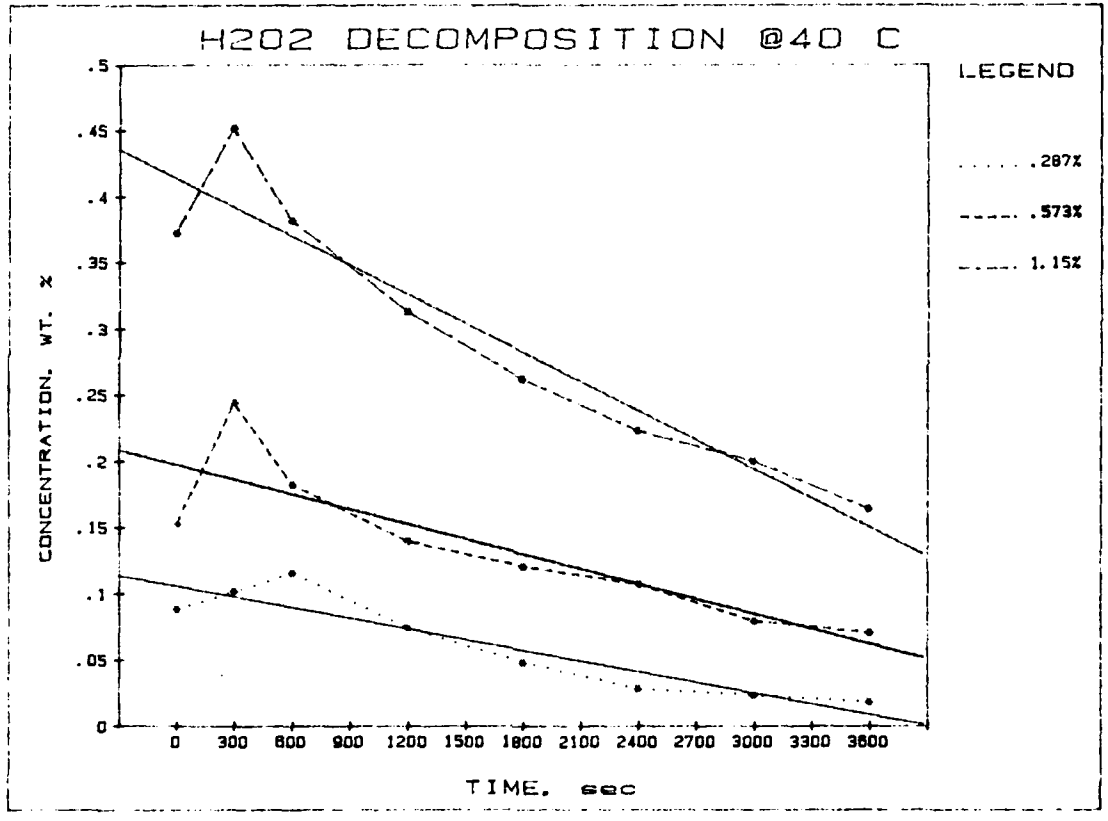

Figure 5. Hydrogen Peroxide Decomposition rates for removing oxide from DU bar for various initial H_2O_2 concentrations. Bath temperature 40°C.

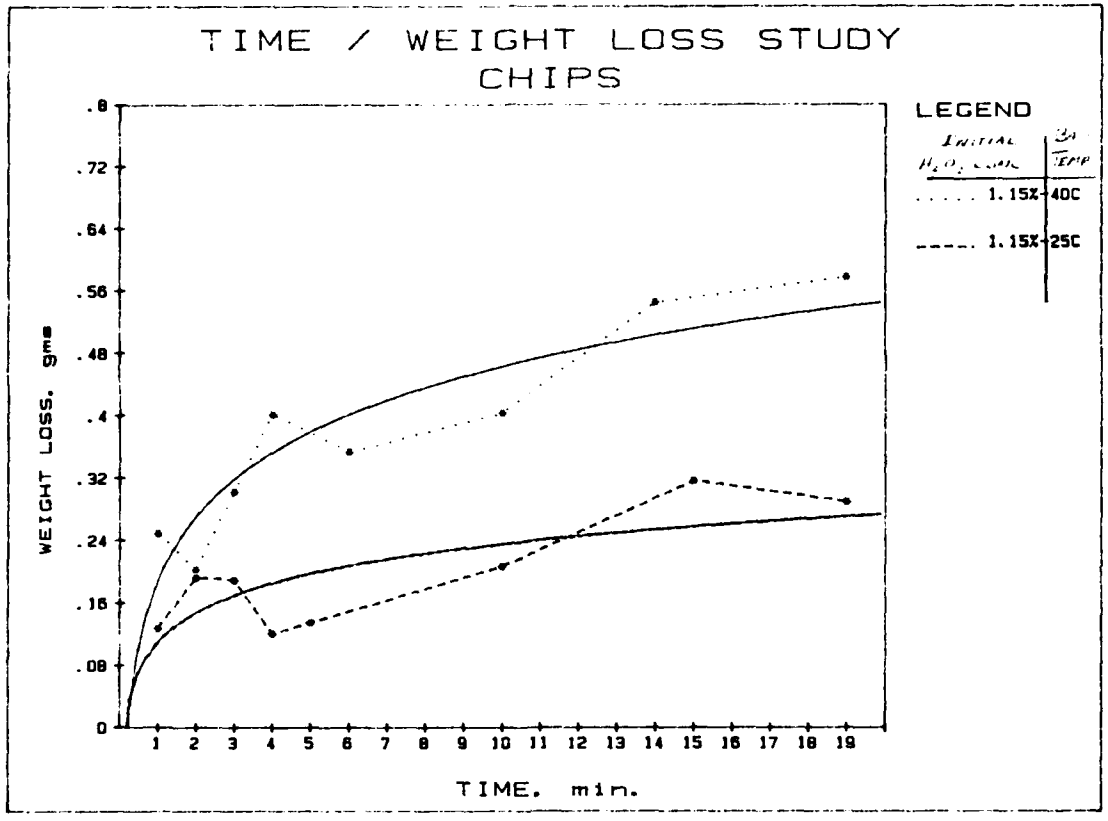

Figure 6. Comparison of chip weight loss due to pickling versus Bath temperature at a constant Bath concentration.

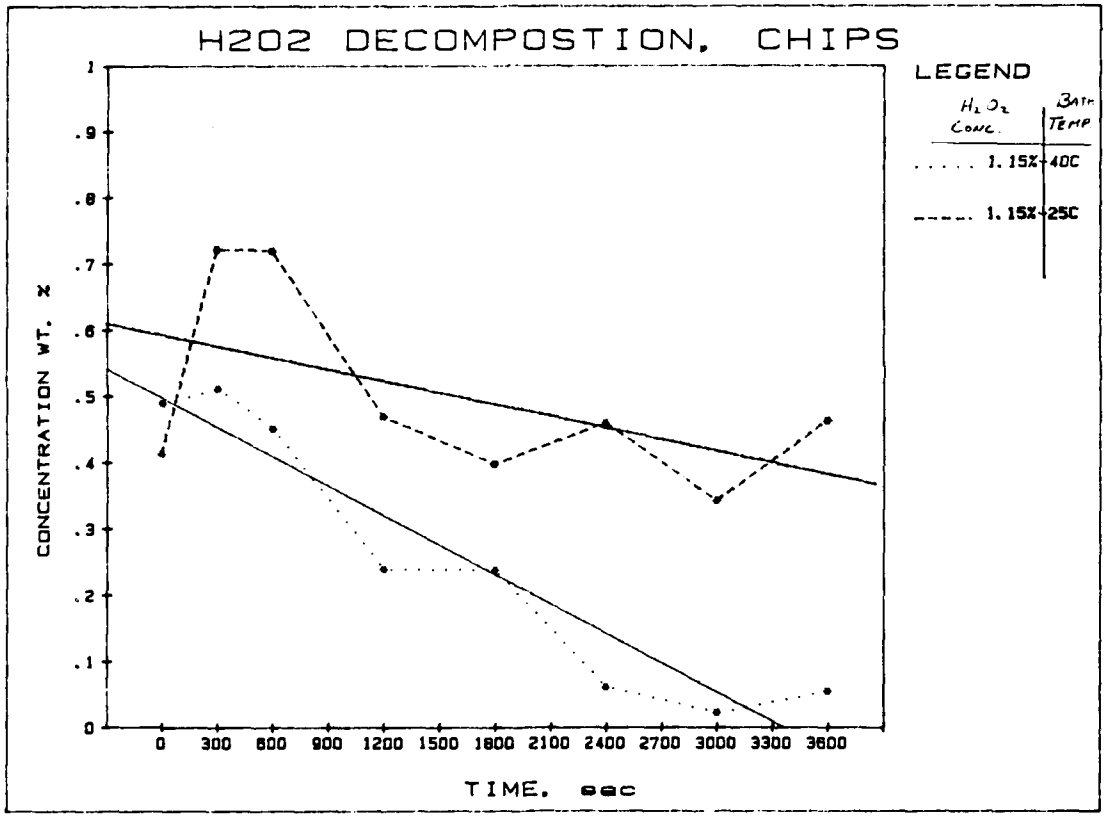

Figure 7. H_2O_2 Bath Decomposition during cleaning of DU chips. Bath temperature was varied while concentration was held constant.

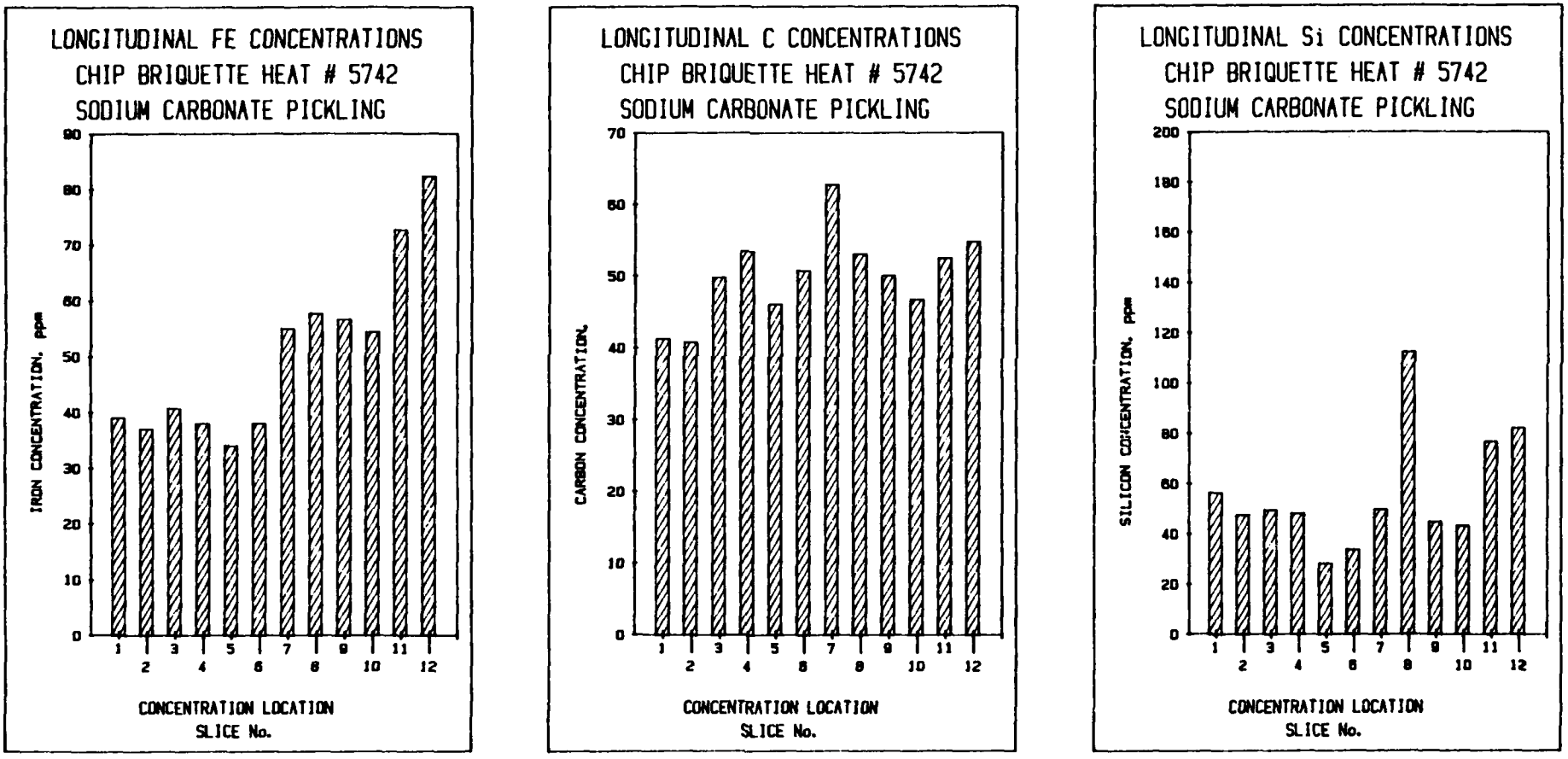

Figure 8. Variation of FE, C and Si concentration through the billet from top to bottom.

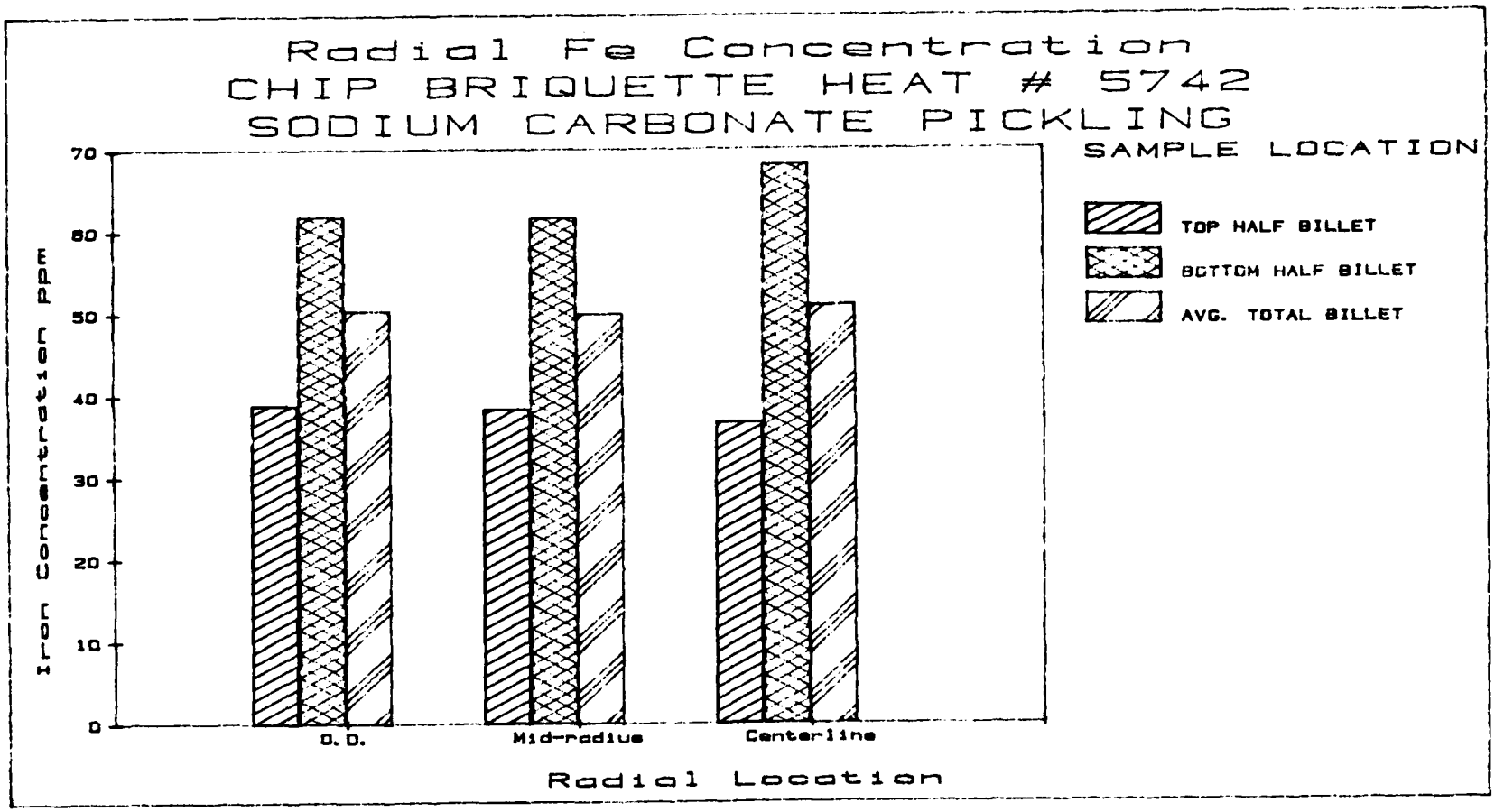

Figure 9. Radial Iron concentration of Heat No. 5742.

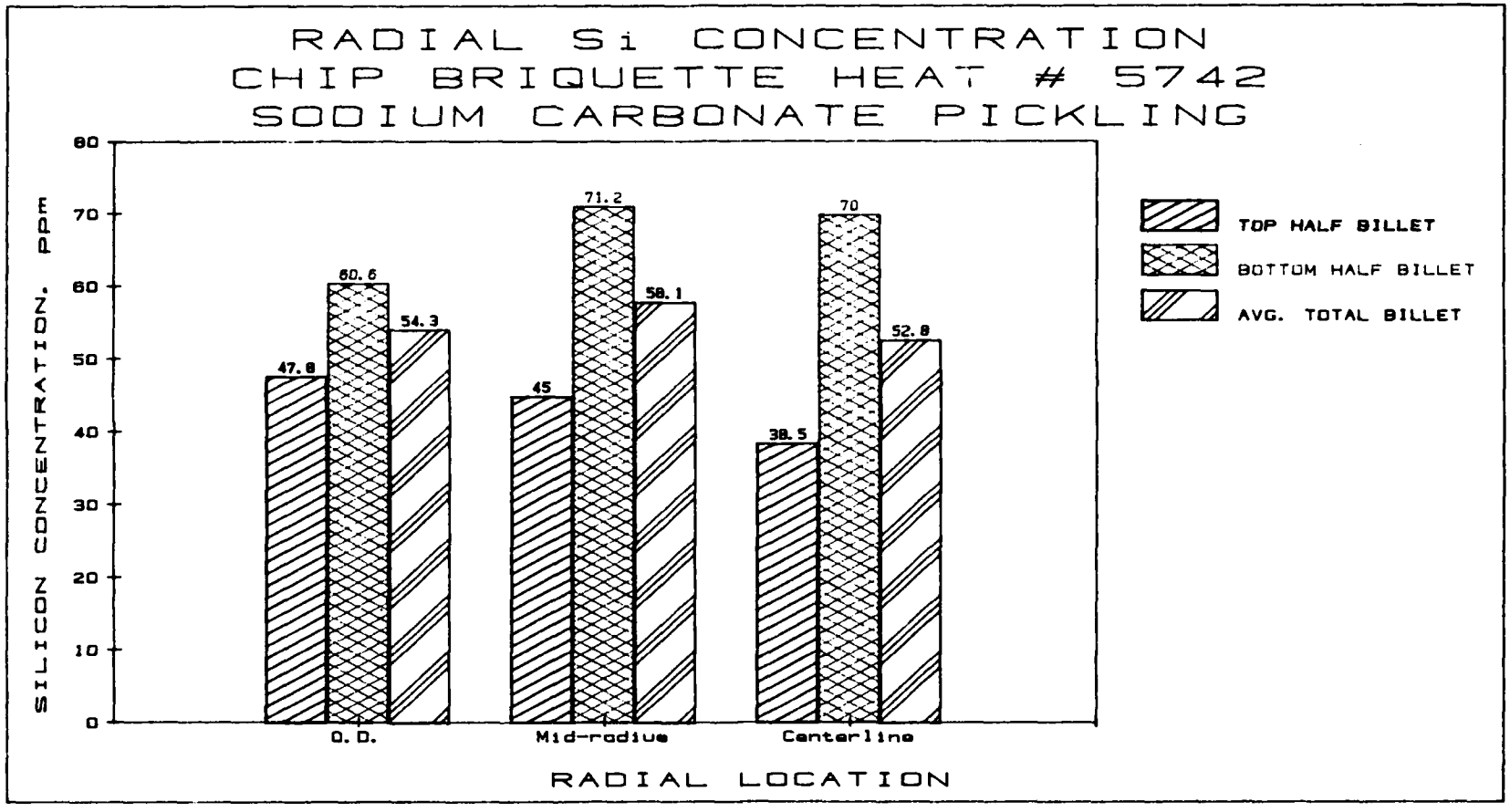

Figure 10. Radial Silicon concentration of Heat No. 5742.

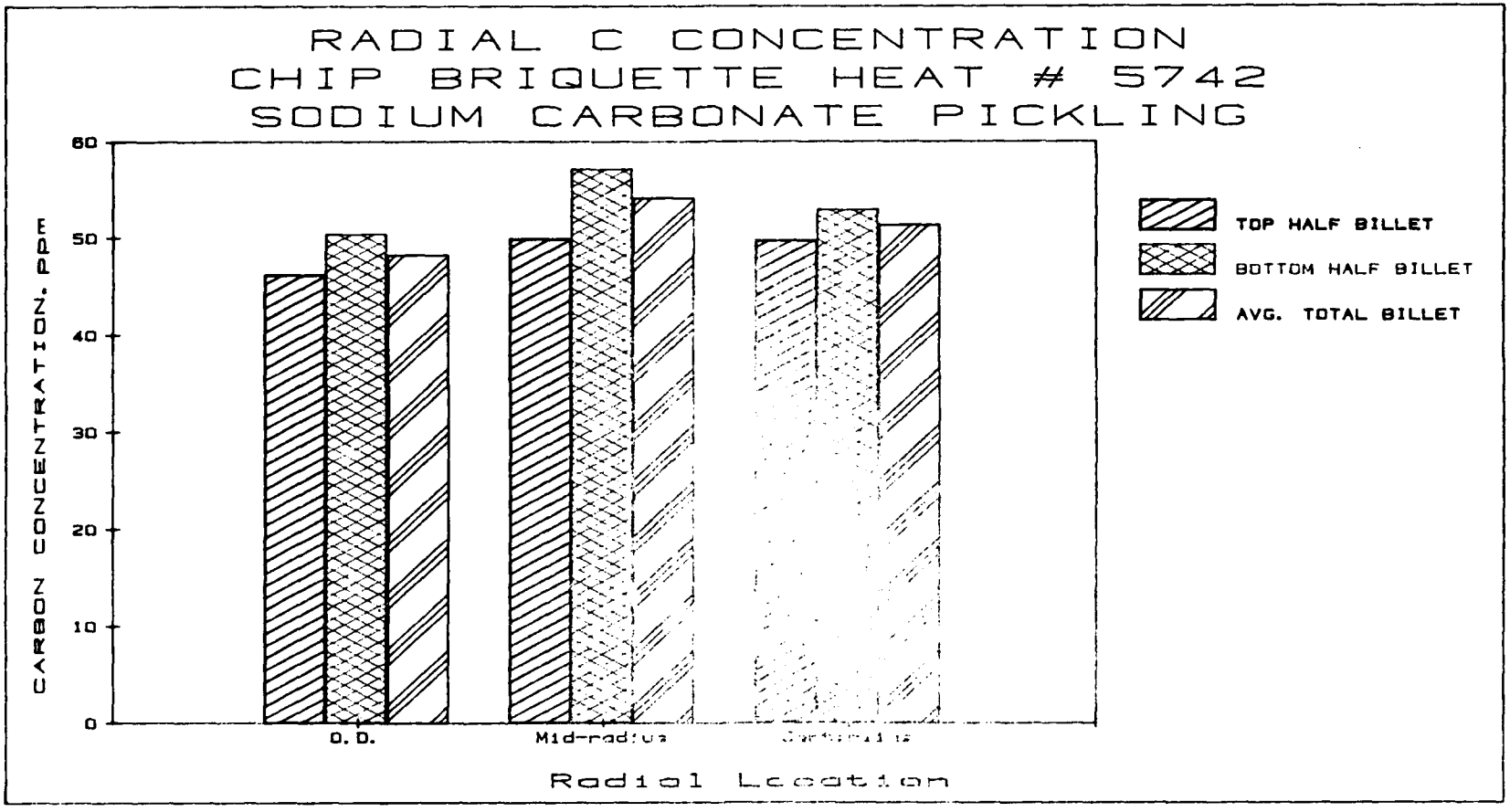

Figure 11. Radial Carbon concentration of Heat No. 5742.

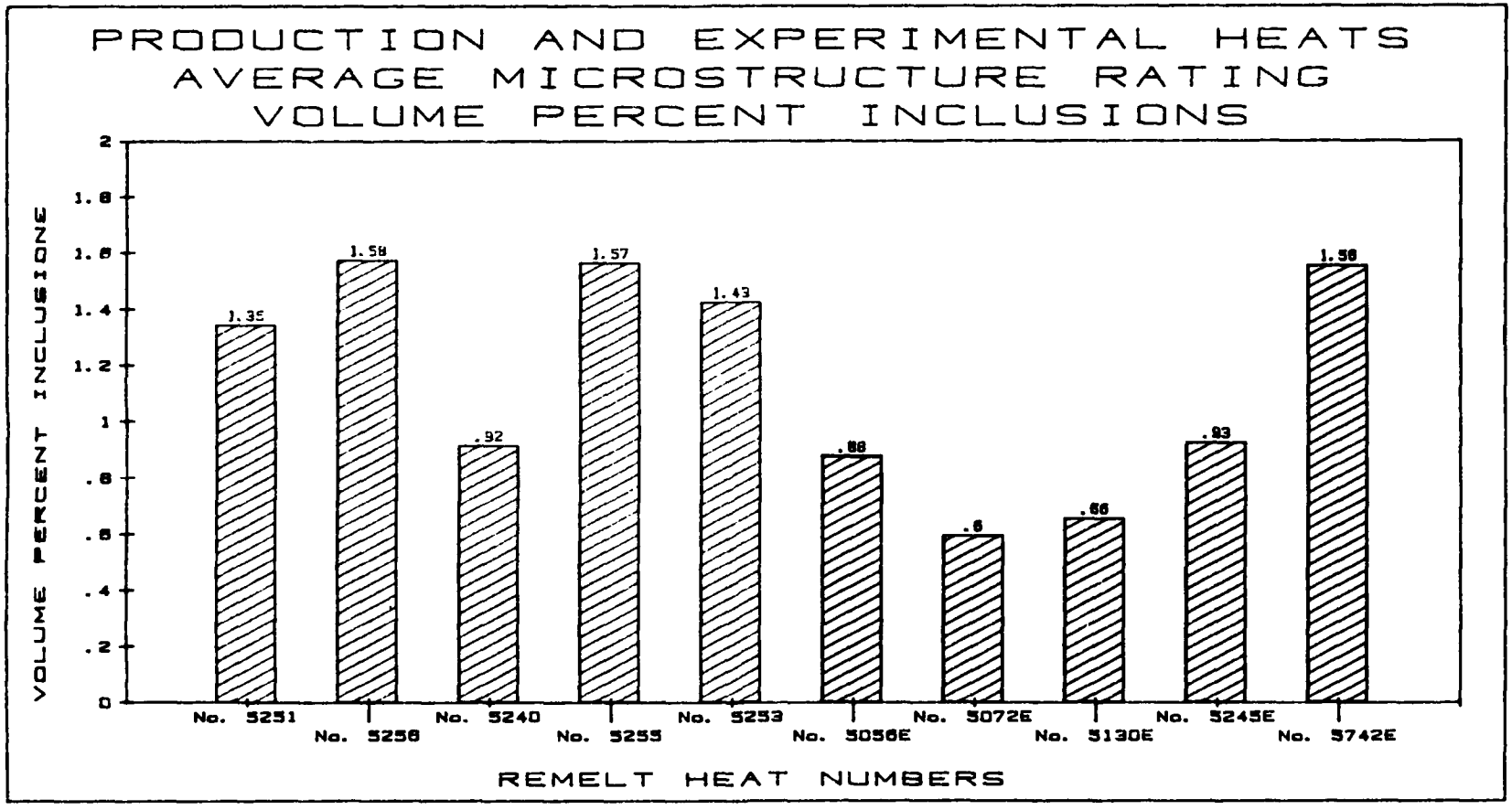

Figure 12. Microcleanliness comparison of Heat No. 5742 and other chip and production heats.

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